# Manufacturing Chemist incorporating MANUFACTURING PERFORM

Vol. XXX No. 7

A PUBLICATION OF THE LEONARD HILL TECHNICAL GROUP

JULY, 1959





# ROTOCUBE MIXING MACHINES

The three-dimensional mixing action of the Rotocube, caused by the rotation of the diagonally mounted cube-shaped drum, is the basis of its outstanding efficiency and rapid action.

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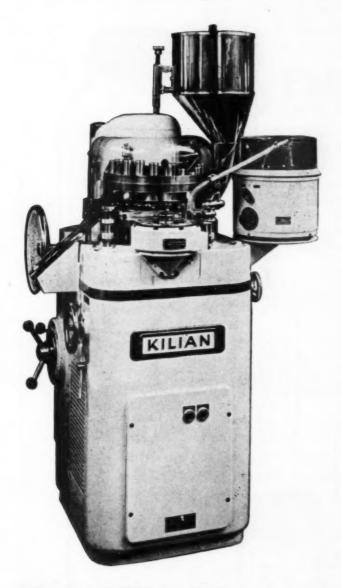
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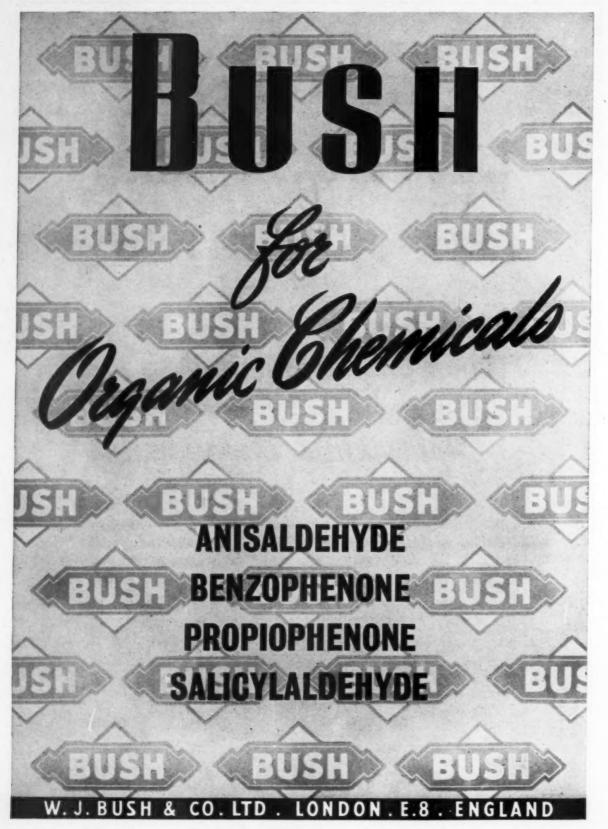
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July, 1959-Manufacturing Chemist



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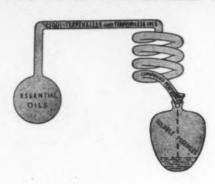
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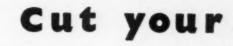
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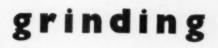
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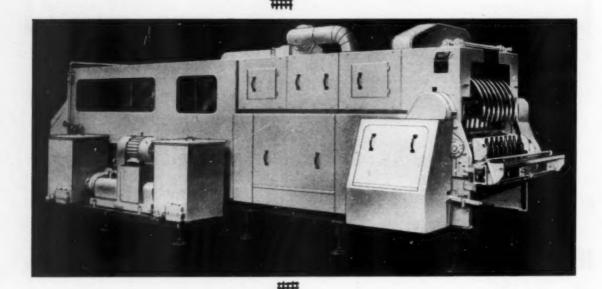
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July, 1959-Manufacturing Chemist



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Pre-determined Counters for counting preset quantities

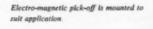
## **Transistor Batch Counters**

This compact unit is used for the counting of articles into predetermined batches. It operates from a perception head which can be either a photo-electric cell and lamp, an electro-magnetic pick-off, or electro-mechanical switch. The counter resets automatically on completion of batch and at the same time an electrical signal is provided which can be used for a variety of functions.

The Batch Counter, fully transistorised, consists basically of an amplifier, the necessary number of dekatron counting units, together with the coincidence and relay units. Built on the individual 'brick' principle which facilitates servicing, the Counter has general reliability and robustness ideal for industrial conditions.

Major overhaul cycle of several years.

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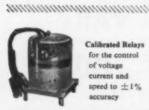
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# Transistor Batch Counters

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Photo-electric cell and lamp arranged for reflected-light operation; this can be varied for interrupted-light operation.





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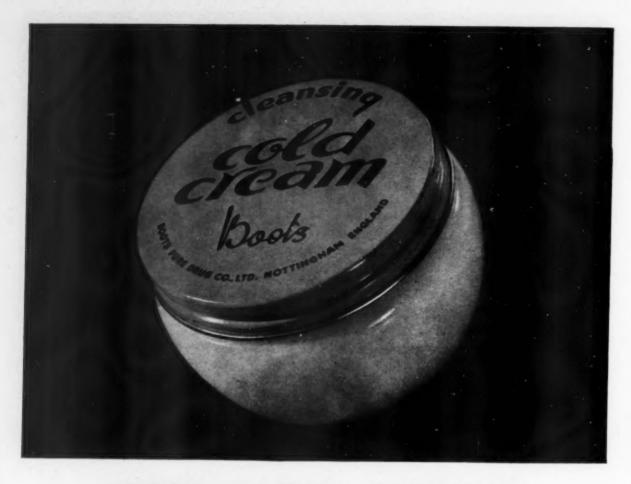
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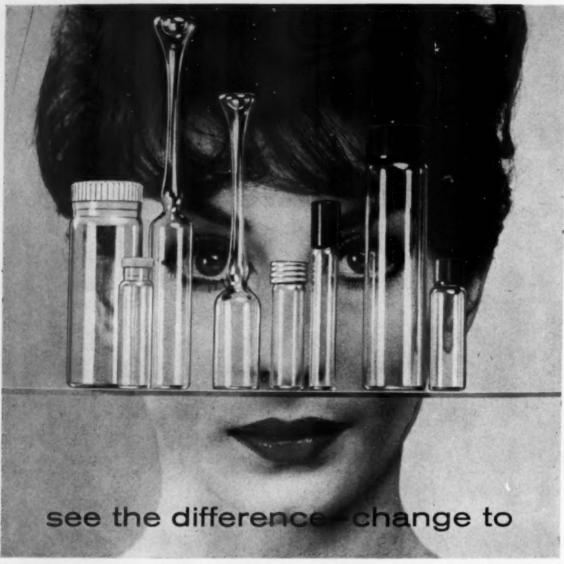
The pack not only looks good-it fits the hand

comfortably, and its flexibility gives an easy controlled flow. The pourer and closure form an integral part of the assembly and were also specially designed for Crosfields (CWG) Ltd. Get in touch with the Plastics Group of Metal Box about a Poly-Tainer for *your* top product.





The Plastics Group of The Metal Box Company Ltd. 37 Baker Street, London, W.1 Telephone: Hunter 5577



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Light, compact and beautifully printed,

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Consult Metal Box Plastics Group-early





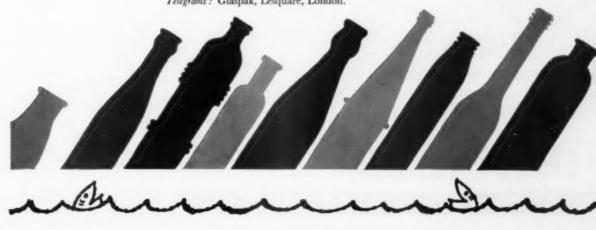
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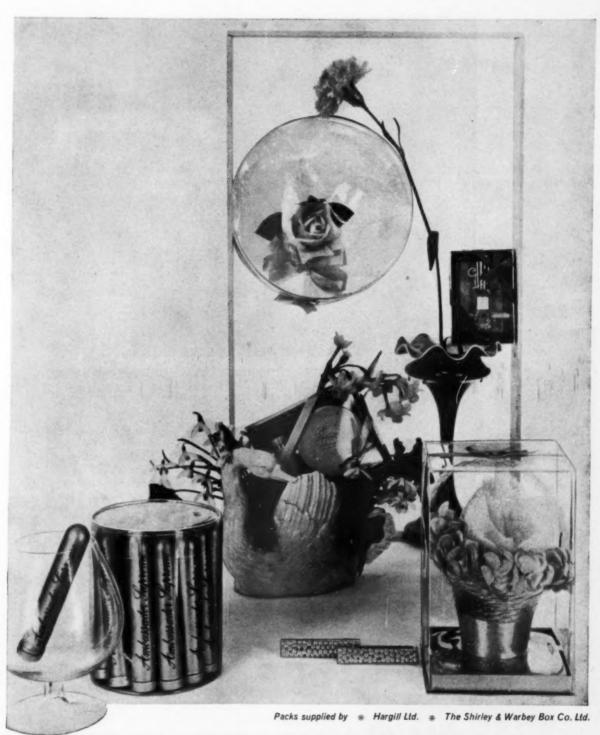
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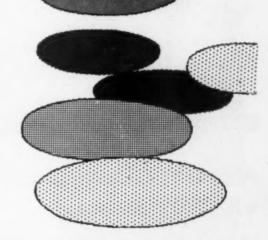
CARBOWAX

POLYETHYLENE GLYCOLS



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For full information including details of grade availability and technical assistance, please contact Section 7-H.



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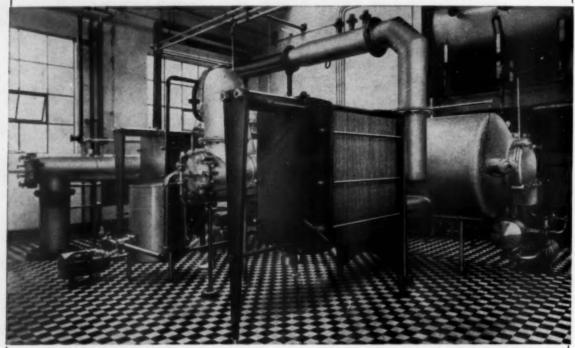








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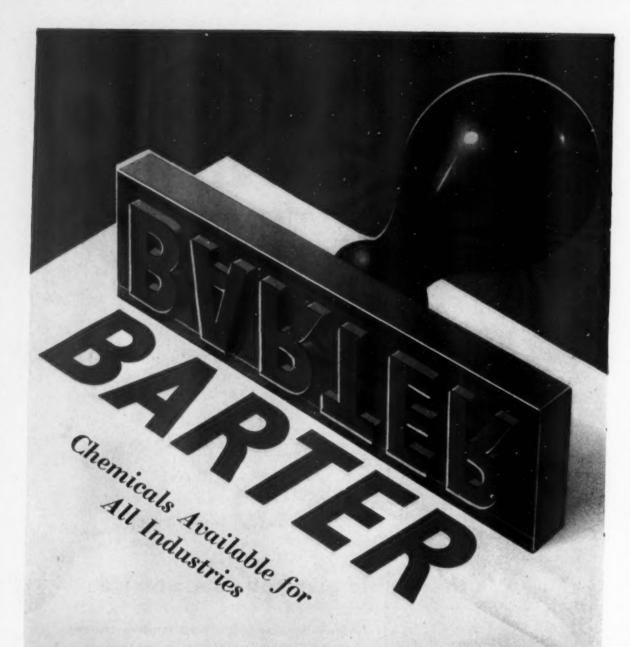
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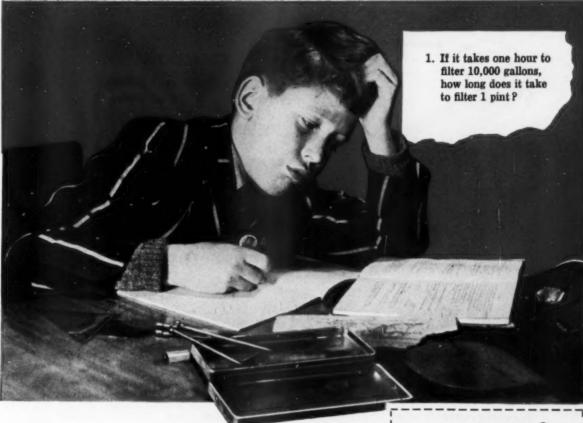




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July Guide

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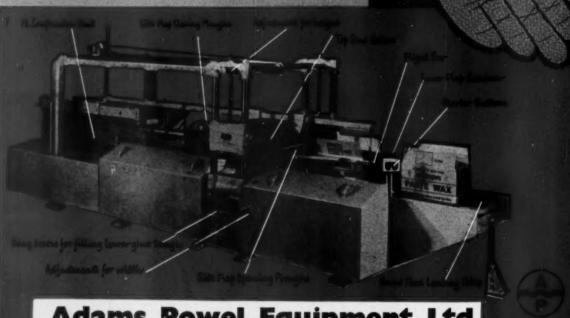
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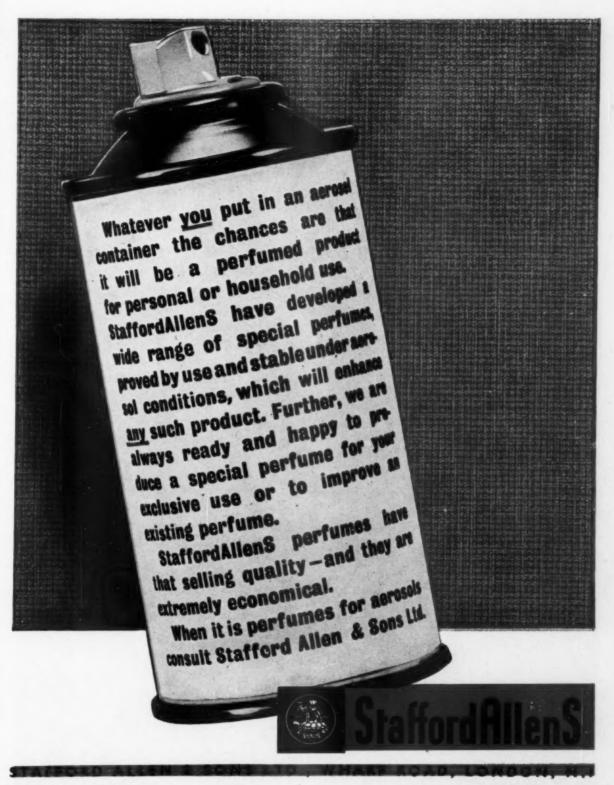
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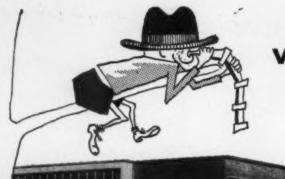
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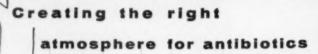
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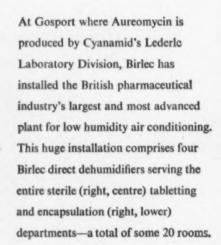






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#### Manufacturing Chemist

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#### Antibiotics in cosmetics

The weight of medical opinion is, quite properly, against the incorporation of pharmacologically active ingredients in cosmetics. The general arguments against this practice can be developed even more strongly in the case of antibiotics, and there will be little surprise at the statement given in Parliament recently to the effect that in this country no cosmetics on the market are known to incorporate antibiotics. Fortunately the control of antibiotics under the Therapeutic Substances Act prevents this kind of misuse.

In the U.S. cosmetic manufacturers are proposing to use bacitracin, neomycin, polymyxin and tyrothricin. They argue that through their use antimicrobial action would be secured against a wide spectrum of micro-organisms and that since these particular antibiotics would rarely be required later on for the treatment of systemic infections the argument that indiscriminate use of antibiotics increases microbial resistance to them does not apply. The Committee on Cosmetics of the American Medical Association oppose these contentions. Although in vitro studies indicate that the development of bacterial resistance to these drugs is not common, there is still the risk that widespread use of them will eliminate the more sensitive bacterial flora, resulting in their replacement by resistant organisms. For instance, there is evidence that the widespread topical use of neomycin may increase the number of patients who develop moniliasis

as a complication of their therapy.

Apart from considerations of drug resistance, sensitivity and toxicity, a strong case against antibiotic-cosmetics is surely the lack of evidence that constant de-germing of the skin (except to reduce axillary odours) is necessarily always or

even frequently desirable. Thus, if the doubtful advantages of dosing cosmetics with antibiotics are set against the increased risk to general public health, the case against such practice seems unanswerable. It is important that this argument be sustained in the U.S., for the cosmetic industry there frequently gives a lead, desirable or otherwise, to cosmetic practices throughout the world.

#### Plastics and medicine

AN UNUSUAL material for the treatment of wounds is expected to be in use soon. It is a powder produced by disintegrating carbamide foam, and advantages claimed for it include one that will certainly be welcomed by the patient; when the powder is used as a dressing it does not stick to the wound.

The powder is said to be non-irritant, to have good adhesion to the skin, and to be capable of bonding twice its weight of water. The particles, which are shaped like lamellæ, are very finely textured and have a surface area said to be 40 times that of the same quantity of starch and 20 times that of talc. The slight acidity of the powder apparently has a favourable effect on the healing of wounds. The carbamide foam itself may also be used as a dressing in the form of thin layers which are placed directly on the wound.

In view of the good compatibility which many plastics have with the human skin, they have been used in surgery for a considerable time to replace bones and joints even in cases of serious surgical operations. Surgeons have implanted plastics replacement parts, to some extent in desperate cases where the amputation of a complete body extremity appeared to be the only possible course.

Surgery requires plastics which combine good physiological compatibility with high mechanical strength, and of interest in this respect, are the new developments in the plastic field, such as the so-called stereo-specific polymers and the technical perfecting of the polycarbonates. Both are new groups of plastics and give rise to great expectations with regard to temperature stability and strength. Polyamide plastic implantations and plastic eye prostheses eyeballs, suture materials, netting, tapes, and even whole arteries have already become established aids in surgery and medicine.

In orthopædics, too, plastics are extensively used. Examples are supporting corsets for immobilisation of the spinal column and corrective collars. Medical trays which are sterilisable in moist heat up to 140°C., Fergusson's specula, bidet basins, etc., are available in a variety of types. A recent plastic instrument is the infusor. In this a hermetically sealed plastics cover contains an ampoule made from chemically pure polyethylene without additives, and a piece of transparent PVC tubing. The ampoule weighs only the twentieth part of a glass ampoule; it is unbreakable and absolutely sterile. The infusor is used only once. Nevertheless, it is able to compete with the conventional instruments because it has been possible to keep its price correspondingly low. PVC tubing has also given best results in tubes for stomach and intestines which can be repeatedly cleaned by boiling in water.

#### Chemicals in sausages

At the time of the Suez crisis in 1956 the idea was put forward of overcoming the shortage of tankers by towing oil cargoes in sausage-like floating containers. This seemingly bizarre proposal has now become reality. Unfortunately for the inventors, the shortage of tankers has given place to a surplus. Luckily for them, however, the idea has attracted the enthusiastic support of I.C.I. and it is to the chemical industry, therefore, that the distinction falls of promoting the first successful deep-sea trial of a flexible, towed container. The containers are called Dracones and the firm responsible for them is Dracone Developments Ltd., which has the support of the National Development Research Corporation, who are exploiting Government-owned patents on the containers.

I.C.I.'s Heavy Organic Chemicals Division became interested in Dracones because they ship abroad large cargoes of lighter-than-water liquids such as plasticising alcohols and solvents, including acetone, isopropyl alcohol and isobutyl alcohol. The cargo chosen for the first trial was a shipment of liquid hydrocarbons from Billingham to Flushing, Holland. Dracone D.4, made of woven nylon fabric proofed inside with oil-resistant acrylonitrile-butadiene rubber and outside with neoprene, and with a capacity of 40 tons, was sent to Billingham by

lorry, unwound from a reel, and filled as it lay in the water at Billingham wharf. After a trial in the open sea, it was hitched to an ocean tug, Fiery Cross, and left for Flushing on May 23. The 260 nautical mile voyage was completed at an average speed of 6.8 knots and the Dracone berthed at Flushing on May 25. The trip through the short, steep seas of the North Sea provided a severe test which the Dracone passed satisfactorily, sliding over and through the wave crests with the minimum of fuss. All night, the acetylene light on the tailfloat of the Dracone shone clearly through the spray, marking her position for the benefit of other shipping.

This historic trial of the sea-going sausage gives hope that a new and economical method of transporting bulk chemicals is at hand. It should be possible for a normal cargo vessel to tow the containers without materially increasing fuel costs, or reducing speed or conventional payload.

#### Match maker

There is more than one Johnnie Walker in our history. One is perpetuated in whisky. A second, born 200 years ago, was a blacksmith's son who took to medicine at 38, graduated at Leyden, and became a pioneer in vaccination when he was made president of a vaccine society. Yet a third, who died 100 years ago on May 1, was the Stockton-on-Tees pharmacist who invented matches named "friction lights" or "lucifers." It is this third holder of a popular name who applied his chemistry to the problem of creating a flame.

Before Walker made a safer form of friction match and thus created demands for potassium chlorate, antimony sulphide and other chemicals, the "oxymuriate match" produced by Chancel in 1805 was typical of the inconvenient, even dangerous, lighters of those days. These "chemical" matches had a head of chlorate, sugar and gum, and were ignited by dipping into a small phial of vitriol. By 1828, when Walker was turning out his "friction lights," the chlorate-vitriol match was much in evidence in Britain, Jones's "Prometheans" being popular. In these a thin spiral of paper had a chlorate igniting composition at the broad end, with a tiny fragile tube of vitriol embedded in the middle, the leather-covered matchbox including two pairs of pliers, one for holding the match and the second for nipping the head. Also about this period Congreve was studying phosphorus in match-heads, "Congreves" together with "Lucifers" sold by Samuel Jones of the Strand being more compelling names than the more practical friction lights of Walker.

Until phosphorus matches were improved, first with Schrotter's red phosphorus championed by Albright of Oldbury and then with phosphorus sulphide, the need had to be satisfied by friction matches invented by the Stockton chemist and copied by others (since Walker refused to patent a public boon). At his High Street premises he dipped

his wooden splints made by part-time labour, selling his first boxes in 1827 at 1s. 2d. a box. Each box included a folded piece of sandpaper for striking. Hundreds of boxes were sold before Isaac

Holden also produced friction matches.

While certain histories of matches refer to Walker's matches as Lucifers, one or two chemically minded enquirers into early samples described in the *Chemical News* the use of slow-burning sulphur by Jones for solving the problem of communicating heat to the wood before the composition burned away. These early matches made absorbing chemical studies as, for example, the use in Germany of magnesia and chalk to neutralise the deliquescent oxidation products of phosphorus in the rival type of igniting composition being produced in Germany.

John Walker goes down in history as one more pharmacist who became in a small way a manu-

facturer.

#### Not Newtonian

NON NEWTONIAN fluids, which crop up in such diverse forms as drilling muds and toothpaste, include also many important plastic materials, rubber solutions, paints, clay suspensions, foodstuffs such as mayonnaise and liquid chocolate, and industrial slurries and emulsions. The laws governing the flow of these fluids are the subject of much interesting research by rheologists, but to the chemical engineer a real move to establish the precise pattern of their behaviour is long overdue. Some day, it is hoped, someone will evolve a reliable general equation for the calculation of non-Newtonian fluid viscosity and thus earn the undying gratitude of countless plant designers. For non-Newtonian flow is a problem that has to be grappled with not only in the design of pipeline systems and heat exchangers but also in such operations as mixing, and although D. F. Riley has commented encouragingly on recent work on the mixing of suspensions and pastes (Chemical and Process Engineering, 1959, 40 (2), 45) co-operation between chemical engineers and physicists on this subject has not been noticeably sustained or fruit-

The issue is confused by conflicting notions as to the nature of non-Newtonian fluids and the correct mathematical approach to dealing with them. A welcome contribution to clearer thinking on the subject is a paper by Dr. D. R. Oliver in the Birmingham University Chemical Engineer (1958, 10 (1), 20) in which he bravely attempts a firm definition and classification of non-Newtonian materials. Dr. Oliver divides these materials into (a) time-independent fluids, in which the shear stress is a function of the rate of shear and nothing else; (b) time-dependent fluids, in which the shear stress rate of shear relationship is a function of time of shear or the previous history of the sample; and (c) viscoelastic fluids in which the property of elasticity is added to the usual viscous properties of

the fluid. These broad groups are again subdivided into a number of smaller groups. Dr. Oliver goes on to present formulæ for the volumetric flow-rate of time-independent, non-Newtonian fluids in tubes of circular cross-section, and also equations for a more general approach to pipeline design.

Rheology

Some interesting contributions of the rheologists to a better understanding of plastic flow are outlined in a paper which was presented to the Royal Society of Arts recently by Dr. E. G. Richardson and Dr. G. W. Scott-Blair. The problems of rolling dough in the bakery the "spreadability" of butter and cheese and the viscosity of biological secretions in animals (measurement of which can yield important data about the physiological condition of the animal) are subjects which come within the range of this branch of science. And, to come back to chemical engineering, so does the conveyance of powders in fluidised beds, where we have an example of a fluid in which, instead of the matrix being liquid, a gas, usually air, carries particles in suspension.

In case all this was not sufficient to convince us that rheology is much more of a "dark horse" than we suspected hitherto, and that the subject is not locked up in obscure and rather eccentric laboratory tests, we are hurled into the universe at large with the observation that the peculiar geological formations at the Giant's Causeway in Ireland have been ascribed to rheological flows "frozen" into position in past ages. The subject of magneto-hydrodynamics, which deals with the interaction between magnetic fields and viscous flows in, for example, the sun, is also a branch of rheology.

These reflections are of the sort which lead us to an increasing awareness that all paths in science lead to the same end—let us hope that, unlike the subjects of rheological experiments, it will not be a "sticky" end. Probably we shall never know enough about non-Newtonian fluids until all chemical engineers and rheologists are one with the chemists, physicists, biologists and all the other

" ists" in this restless, ignorant world.

#### **Printing Dispute**

We regret that we are compelled to reduce the size of this issue owing to the dispute in the printing industry to which we are not a party and over which we have no control. Normal format will be resumed as soon as possible.

## Chemical Engineering and Process Research at new D.S.I.R. Laboratory

Programme of the £620,000 Warren Spring Laboratory

AT the new Warren Spring Laboratory of the Department of Scientific and Industrial Research at Stevenage, Herts., there are laboratory and pilot-plant facilities for process research and 'velopment over a wide field, not iimited to particular fields of technology. The Laboratory is equipped to tackle all sorts of chemical engineering problems which the research departments of universities and industrial firms cannot (or, in some cases, will not) tackle for themselves.

The Laboratory's programme on distillation supplements that being carried out in the universities under the sponsorship of the A.B.C.M. and B.C.P.M.A. following the report of the Distillation Panel (summarised in our June issue). In addition to its main programme, the Laboratory undertakes to carry out certain research projects on behalf of industry, these being sponsored by the firms concerned.

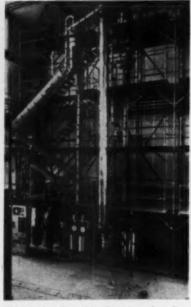
The main target for the Laboratory's pilot-plant investigations at present is the improvement of mineral processing techniques and the synthesis of oil from carbon monoxide and hydrogen (Fischer-Tropsch). These are supported by basic research, including the research in chemical engineering which is being transferred from the National Chemical Laboratory.

#### 4620,000

The Establishment, including buildings, services, fixed laboratory fittings, library bookshelves and all site works, was built for £620,000—equivalent to £6 7s. per sq. ft. The main, three-storey building is 872 ft. long and 37 ft. 6 in. wide, the laboratories being housed within light, demountable partitioning for flexibility. A three-storey administration block runs at right angles to the main building and there are three pilot-scale buildings linked to the main laboratory block by a corridor with small-scale laboratory units on each side.

#### Three tasks

The Chemical Engineering Divi-



The Fischer-Tropsch pilot plant for oilsfrom-coal synthesis.

sion at Warren Spring, headed by Dr. P. H. Calderbank has three tasks:

 (i) Research on physical operations which play an important part both in processes which are under development in the Laboratory, and those in more general use in industry;

(ii) Chemical engineering research sponsored by industry or by Government departments.

(iii) Accumulation of basic information in chemical engineering for use by other sections of the Laboratory and by industry.

As the D.S.I.R. is already carrying out work on heat transfer and fluid flow elsewhere, the Division is concentrating at present on problems of mass transfer, in particular to obtain data which will permit more accurate prediction of the performance of gas-liquid contacting equipment, and thus to facilitate the design of distillation columns, gas-absorption towers, and reactors of the liquid-phase type, of which the Fischer-Tropsch

slurry reactor is a particular example.

Four main lines of study are being followed:

Bubble dynamics. The evaluation of the gas-liquid interfacial area on distillation plates and its relation to the physical properties of the system and the operating variables of the equipment.

Gas-liquid mass transfer rates. Measurement and correlation of the liquid- and gas-phase mass transfer coefficients on distillation plates.

Fluid mixing. Evaluation of the degree of fluid mixing on distillation plates in such a form as to allow this factor to be introduced into the mass-transfer rate equations.

Specific problems in gas absorption with chemical reaction. The prediction of the overall rate in certain specific chemical reactions will involve the study of the reaction kinetics and associated physical rate processes.

Some pilot-scale experiments are at present being carried out in a laboratory originally designed for benches. One item of equipment here is a small distillation column fitted with a brass sieve plate and observation window allowing measurement of the reflectivity of the foam which forms in the column and providing a means of arriving at the interfacial area.

Work is also being carried out on promoters of dropwise condensation for heat exchangers, a study of the mode of action of such promoters being aimed to help in the development of compounds which will be effective for prolonged periods with a wide range of metal surfaces. It is proposed to study the mechanism of synthetic promoters to metal surfaces by labelling with a radiochemical substance (C<sub>14</sub>) and to observe the subsequent history of these substances under conditions of use in heat exchangers.

#### Oils and chemicals from coal

As part of the Ministry of Works' programme to find ways of making more efficient use of fuel in Britain,



The new laboratory was built at a cost of £6 7s. per sq. ft., including buildings, services, laboratory fittings, library bookshelves and all site work. The main three-storey building is 372 ft. long by 37½ ft. wide.

the Laboratory is carrying on with the studies of the synthesis of oils and chemicals from carbon monoxide and hydrogen previously carried out at the Fuel Research Station at Greenwich (which the new Stevenage laboratory replaces).

The development of oils from coal involves (i) complete gasification of coal to yield a mixture of carbon monoxide and hydrogen, and (ii) catalytic conversion of the gas mixture into the desired end-Warren Spring is concerned only with stage ii. Economic considerations indicate that the most promising version of the Fischer-Tropsch process for use under British conditions is the "slurry" process, in which the gas mixture is passed through a suspension of powdered catalyst in molten wax at 250° to 300°C. and at pressures between 5 and 30 atm.

The objectives of the programme are:

- to develop an iron catalyst of longer life and higher activity than those at present available and one that will yield a high proportion of desired end products;
- (2) to select operating conditions (gas composition, temperature, pressure) to combine a high reaction-velocity with a given distribution of end-products;
- (3) to obtain data necessary to design a full-scale reaction vessel.

Synthesis gas is produced on the site at a rate of 4,500 std. cu. ft./hr. in a standard, water-gas generator. There are arrangements for feeding carbon dioxide with the steam so as to vary the hydrogen carbon monox-

ide ratio between 1·15 and 0·6. The gas is purified from hydrogen sulphide in Gastechnik towers and washed with caustic soda to control the carbon dioxide content. It is then compressed to 15 atm. pressure in the first two stages of a four-stage compressor, passed through the active carbon scrubbers to remove organic sulphur compounds, and then further compressed in the third and fourth stages to a pressure of 120 atm. The gas plant was designed and erected by Humphreys and Glasgow Ltd.

In addition to the Fischer-Tropsch pilot plant there are six bench-scale units operated by the Process Development Division of the Laboratory for the testing of catalysts, treatment of catalysts, etc. The Fischer-Tropsch pilot plant takes up to about 3,000 cu. ft./hr. of synthesis gas.

#### Cleaner air

In addition to its mineral processing and process and develop-ment work the Laboratory will carry on the research on atmospheric pollution begun at Green-One interesting line of research was concerned with the removal of sulphur from flue gases. The Fuel Research Station at Greenwich investigated proposals to scrub flue gases with ammonia solutions and recover the sulphur as ammonium sulphate, and found process practically economically feasible. The central Electricity Generating Board and Simon-Carves Ltd. completed demonstration work on a large unit and confirmed the favourable cost estimates. Supplies of ammonia and the demand for ammonium sulphate limit the application of this process. Other proposals have therefore been investigated; for instance, involving the recovery of sulphur as sulphuric acid.

Chemical Engineers' regulations. The latest edition of the regulations of the Institution of Chemical Engineers contains guidance on training and experience requirements for associate membership, and also the new syllabus for the engineering drawing paper of the institution examination which is to come into force in 1960.

E.R.A. publications. The British Electrical and Allied Industries Research Association has published two technical reports. One covers the determination of the relative ease of ignition, by the sparks produced when an inductive current is broken, of the vapour of isohexane, while the other reviews published information on the incendivity of sparks from light alloys in combustible atmospheres met with in industry. They are priced at 6s. 6d. and 12s. 6d. respectively.

Glycerin data sheet. A new sheet summarises the physical properties of glycerin, including short tables showing specific gravity, freezing points and vapour pressure/relative humidity data for different concentrations of glycerin in water.

The sheet also describes the three principal grades of refined glycerin, namely: Chemically pure, which conforms to both B.S. 2625 and the current British Pharmacopæia. Technical grade, which conforms to B.S. 2623, and dynamite grade, which conforms to B.S. 2624 and is refined specifically for use in explosives manufacture.

The brochure is available from the Secretary, The United Kingdom Glycerin Producers Association Ltd., 5 Bridewell Place, London, E.C.4.

#### The Search for Insulin Substitutes

By R. B. Hunter,\* M.B.E., M.B., F.R.C.P.E., M.R.C.P., D. M. Shepherd,\* B.Sc., PH.D., and J. M. Stowers,\* M.A., M.D., M.R.C.P.

There are probably few of us who are not acquainted with someone who suffers from diabetes mellitus, commonly referred to simply as diabetes, which when untreated is a distressing condition characterised by excessive thirst, excessive excretion of urine, and by the presence of abnormally large quantities of glucose in the blood (hyperglycamia) and in the urine (glycosuria). Diabetes has been known for a very long time. A case of what was almost certainly diabetes was described in an Egyptian papyrus of about 1500 B.C. The name itself, introduced by the Greek physician Aretaeus of Cappadocia (A.D. 30-90), means to pass through or to siphon, and is an apt description of the characteristic inability of the victim to retain water. It was a further 1,800 years or so before the essential nature of the disease was elucidated. However, in 1922, when the work of Banting and Best had shown that diabetes arose from a deficiency of the secretion of the insulin-producing  $\beta$ -cells of the pancreas, insulin extracted from mammalian pancreas became, and still remains, the standard preparation for the treatment of diabetes. The greatest disadvantage of insulin is that it must be administered over a period of many years by daily injection, with all the accompanying discomfort and inconvenience which this entails. It is for this reason that research has recently been intensified towards the development of synthetic drugs which, taken by mouth in a convenient tablet form, lower the blood sugar (hypoglycamic effect), prevent glycosuria, and alleviate the acute symptoms of diabetes.

WHILE many substances have been observed to lower the blood sugar level of man and laboratory animals, the present discussion is confined to the main classes of synthetic compounds which are, or which promise to be, of practical therapeutic value.

#### Guanidine derivatives

The hypoglycæmic effect of guanidine (1) was first systematically studied by Watanabe¹ who found, however, that this compound produced neurotoxic side-effects. Interest in guanidine derivatives was further stimulated by the identification of Galegine (2), the hypoglycæmic principle present in the seeds of Galega officinalis, as isoamylenyl-guanidine by Späth,² and of Agmatine (3), a hypoglycæmic constituent of herring sperm, as 1 - guanidino - 4 - aminobutane by Kossel.³

Subsequently Frank and coworkers 4, 5, 6, 7 found that a more intense hypoglycæmic action was exhibited by compounds having a polymethylene chain interposed between two guanidine molecules. In this way decamethylenediguanidine (4, n=10) and duodecamethylenediguanidine (4, n=12), known as Synthalin A and Synthalin B respectively, were developed during the period 1925-30.

In spite of their pronounced hypoglycæmic activity these substances were regarded as unsuitable for long-term use as insulin substitutes, since, in therapeutic doses, they produced unpleasant side-effects such as irritation of the gastric mucosa, nausea, vomiting and damage to the kidneys and liver. §, §, 10

When the guanidines were also found to possess useful antitrypanosomal activity, their study as chemotherapeutic agents was continued for many years, but they were no longer regarded as potential antidiabetic compounds. Recently, however, guanidine derivatives of a somewhat different nature from Synthalin have shown promise as hypoglycæmic drugs. Research in

Department of Pharmacology and Therapeutics,\* and Department of Medicine,† Queen's College, Dundee, University of St. Andrews. this field has thus received a fresh stimulus, and several substituted diguanidines, especially the compound known as DBI,  $\beta$ -phenylethyldiguanide (5) have been subjected to clinical trial.<sup>11</sup>

No serious side-effects appear to have been reported following the long-term administration of DBI to patients, and it is possible that the toxic hazards of guanidine derivatives have been overestimated. Recent work<sup>12</sup> with 1:1-dimethyldiguanide (6), a compound first studied about 30 years ago, <sup>13</sup> indicates that its toxicity is so much less than was originally

believed that it can now be successfully administered to human diabetics. Further support for the view that exogenous compounds containing the guanidine group are not necessarily toxic is found in the case of Paludrine (N-(p-chlorophenyl)-N'-isopropyldiguanide) (7) which

has been used successfully for some years in the treatment of malaria. It is pertinent to note here that Paludrine also possesses mild hypoglycæmic acitivity, 11 although this is insufficient to render it suitable for the treatment of diabetes.

Hypoglycin A

A completely new approach to the problem of finding an orally active antidiabetic drug has been suggested through studies of the toxic factors in the fruit of Blighia sapida, a tree which grows abundantly in the West Indies to which it was introduced from West Africa. Ingestion of the fruit, which is regularly eaten by natives in these areas, can produce toxic symptoms and occasionally death. Two of the toxic substances, isolated in 1954 by Hassall, Reyle and Feng,14 were found to cause severe hypoglycæmia, depletion of liver glycogen, and death; these compounds were accordingly named Hypoglycin A and Hypoglycin B. The former, which is much the more active and abundant, has recently been identified by several independent groups of workers15.20 as an aminoacid of relatively simple, but unusual, structure, viz. α-amino-β-[1-(2 - methylenecyclopropyl)] - propionic acid (8).

The activity of Hypoglycin A appears to be associated with the terminal ethylenic linkage, since reduction of this group results in the disappearance of the hypoglycæmic action. Furthermore, Anderson and coworkers<sup>19</sup> have synthesised three compounds which have hypoglycæmic activity of the same order as Hypoglycin A, but, although all three contain a terminal ethylenic bond, only one of them is an amino-acid. The substances in question are 2-amino-5hexenoic acid (9), 4-pentenoic acid (10), and 3-methylenecyclobutanecarboxylic acid (11).

Anderson et al. point out that the ethylenic group is separated by two carbon atoms from the carboxyl group in compounds (10) and (11) or, in the case of the amino-acid (9), from the potential carboxyl group which one would expect to be formed in vivo by oxidative deamination and decarboxylation. If this were indeed a structural requirement for hypoglycæmic activity in this class of compound, it would account for the absence of activity in 2 methylenecyclobutane-carboxylic acid (12), in which the

ethylenic group is separated by one (or three) carbon atoms from the carboxyl group.

The synthesis of Hypoglycin A has recently been reported, 21 and it is to be hoped that further synthetic and pharmacological studies will reveal related compounds, some of which may prove sufficiently promising to justify clinical trial in the treatment of diabetes.

#### Sulphonamides

For more than 20 years sulphonamides of the general formula (13)

have been used in the treatment of certain bacterial infections; R may be one of a wide range of alkyl, aryl or heterocyclic groups. In 1942, when treating cases of typhoid fever with a newly synthesised sulphonamide, 2 - (p - aminobenzenesulphonamido) - 5 - isopropyl-thiadiazole (14, R=iso-Pr),

Janbon<sup>22</sup> observed typical hypoglycæmic reactions which he attributed to the drug.

This observation was quickly confirmed, and Loubatières 23 on the one hand and Bovet and Dubost<sup>24</sup> on the other simultaneously and independently studied the effect on biological activity of varying the group R in formula 14. results, which were in good agreement, indicated that the hypoglycæmic action was maximal when  $R = n - C_4H_9$ ;  $i - C_4H_9$ ;  $t - C_4H_9$ ;  $n - C_5H_{11}$ . Slightly lower but acceptable activity was observed when  $R = n - C_3H_7$ ;  $i - C_3H_7$ ;  $i - C_5H_{11}$ . Compounds in which hypoglycæmie activity was low or nonexistent were those having R=CH3; C2H5; n-C<sub>6</sub>H<sub>13</sub>; n-C<sub>7</sub>H<sub>15</sub>; C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>;

Other inactive compounds were 2 - amino - 5 - isopropylthiadiazole (15), 2 - benzenesulphonamido - 5-isopropylthiadiazole (16), and 2-(p-aminobenzoylamino - 5 - isopropylthiadiazole (17).

It appears, therefore, that in this series of compounds hypoglycæmic activity is critically related to the presence of the p-aminobenzene-sulphonamido group, i.e. the active compounds are all derivatives of sulphanilamide; the size of the alkyl group R is also important but is less critical.

A further compound of this type, 2 - sulphanilamido - 5 - cyclopropylthiadiazole (14; R=—CH——CH<sub>2</sub>)

now assumes some theoretical interest in view of its slight structural relationship to Hypoglycin A, both compounds possessing the cyclopropane ring system; although it showed strong hypoglycæmic activity in normal rabbits, it was actually markedly hyperglycæmic in alloxan diabetic animals. 25

From the therapeutic point of view the sulphonamidothiadiazoles proved disappointing because of their relatively high toxicities.

Sulphonylureas

In 1954 Franke and Fuchs<sup>26</sup> in Germany had an experience similar to that of Janbon twelve years earlier, when they found that a new sulphanilamide derivative synthesised as a potential chemotherapeutic agent produced marked hypoglycæmia in non-diabetic patients. In the first reports<sup>26</sup>, <sup>27</sup>, <sup>28</sup> the new drug, N-(p-aminobenzene-sulphonyl) - N' - butylurea (18), was referred to as BZ 55, but it is now commonly called Carbutamide.

Soon after the announcement of Carbutamide the first reports appeared, <sup>29</sup> also from Germany, of a closely related sulphonamide, N - (p - toluenesulphonyl) - N' - butylurea (19), originally designated D860, but now known as Tolbutamide.

Carbutamide, conforming as it does to the general formula (13), is an antibacterial as well as an antidiabetic substance. Tolbutamide, which differs from Carbutamide only in having the p-amino group replaced by a p-methyl group, is therefore devoid of antibacterial action, but the hypoglycæmic activity remains unimpaired. Thus the presence of the p-aminobenzenesulphonyl group is not a prerequisite for antidiabetic activity among the sulphonylureas as it was in the case of the sulphonamides discussed above.

The detailed structure-activity relationships of the sulphonylureas represented by the general formula (20) are now being eluci-

dated. 30, 31, 32 While R may be varied between fairly wide limits embracing alkyl, aryl, and cyclic alkyl substituents without the hypoglycæmic activity being completely lost, peak activity is attained when R is n-propyl or n-butyl. Modifications of the urea part of the molecule to give, for example, sulphonylthioureas or sulphonylcarbamates, diminish the activity. The group Ar may consist of a substituted phenyl, naphthyl or heterocyclic radical, the hypogly-cæmic activity of the resulting products varying from very weak to very strong. In general, the compounds have maximal activity and duration of action when Ar is a para-monosubstituted phenyl group, particularly when the para-substituent is chlorine.

On the basis of these studies N - (p - chlorobenzenesulphonyl)-N'-(n-propyl)-urea (21) has been developed as the most potent and longest-acting hypoglycæmic sulphonylurea at present available, and it is now undergoing extensive clinical trial under the name of Chlorpropamide.

Recently it has been reported <sup>33</sup> that N-alkylsulphonyl-N'-butylureas (22), in which the group Alk is a higher alkyl group, are also hypoglycæmic substances.

Two main methods are available for the synthesis of sulphonylureas:

(1) The appropriate sulphonamide

 The appropriate sulphonamide is treated with an isocyanate in the presence of alkali;<sup>34</sup>

$$\begin{array}{c} R \longrightarrow SO_3 \longrightarrow NH_3 + O = C = NR' \longrightarrow \\ R \longrightarrow SO_3 \longrightarrow NH \longrightarrow C \longrightarrow NHR' \\ 0 \end{array}$$

(2) The appropriate sulphonamide is reacted with an alkyl chlorocarbonate in the presence of alkali to give a carbamate<sup>31</sup>, <sup>32</sup>:

$$\begin{array}{c} R-SO_{s}-NH_{z}+CI-C-OR'\longrightarrow\\ \parallel\\ R-SO_{s}-NH-C-OR'+HCI\\ \parallel\\ \end{array}$$

On treatment with an alkylamine the carbamate yields the required sulphonylurea.

$$\begin{array}{c} R-SO_{s}-NH-C-OR'+R''NH_{s}-\rightarrow\\ \parallel\\ O\\ R-SO_{s}-NH-C-NHR''+R'OH\\ O\\ \end{array}$$

Salicylates

The hypoglycæmic activity of salicylates has been known since 1875. Thus, prior to the introduction of insulin, aspirin (acetylsalicylic acid, 23) was occasionally used as an oral hypoglycæmic agent, though it did not come into general clinical use on account of the large, frequently toxic, doses required. It is only recently that salicylate therapy has been re-investigated with promising results.

In vitro experiments35 suggest that the most strongly hypoglycæmic salicylates are those which interfere with the metabolic process known as oxidative phosphorylation; aspirin and 2: 3-dihydroxybenzoic acid (24) fall into this On the other hand category. salicylate derivatives such as 2:5dihydroxybenzoic acid (gentisic acid, 25) which fail to uncouple oxidative phosphorylation, appear to have little or no hypoglycæmic action.36 In man the principal studies have been carried out with sodium salicylate and aspirin, but the effects of other salicylate derivatives are now under investigation.

#### Mode of action

Diabetes is due to a deficiency of insulin which may be absolute or relative. Insulin would seem therefore on first principles to offer the most satisfactory treatment for diabetes but it has the disadvantage that it has to be injected. No oral anti-diabetic drug could do better than simulate the action of insulin as closely as possible, and it is therefore appropriate to consider the mode of action of these oral compounds against that of

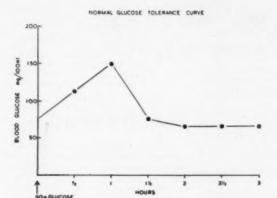


Fig. 1. Normal glucose tolerance curve.

insulin as a standard reference. In spite of nearly 30 years of work there is still controversy about the exact mechanism of insulin's action and in particular whether its main activity is in the liver or in the muscles and other peripheral tissues. In summary insulin is known to increase the permeability of cells to glucose, to increase the glycogen content of muscles and liver and to increase their utilisation of oxygen. A good deal is known about the biochemical reactions which are stimulated by insulin within the cell, leading to an increase of aerobic glycolysis and a build-up of stores of energy-rich phosphate.

The most practical approach to the aim of obtaining hypoglycæmic drugs which simulate the action of insulin is to use compounds which stimulate the insulin-producing Bcells of the pancreatic islets in those diabetics who still possess viable β-cells. There is a good deal of evidence to suggest that the hypoglycæmic sulphonylureas have such an action, and this would explain why they are ineffective in pancreatectomised animals and in completely insulin - deficient human diabetics. The hypoglycæmia achieved by most of the other anti-diabetic compounds considered above does not seem to be dependent on such a mechanism of increased There are insulin production. many possible ways in which the blood sugar could be lowered and which could constitute the mode of action of hypoglycæmic drugs. Thus they could increase the utilisation of glucose in the muscles as do the diguanides and salicylates. These compounds share with insulin their ability to increase the permeability of the cell membrane to

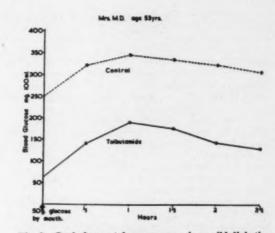


Fig. 2. Oral glucose tolerance curves in a mild diabetic.

— — — On no treatment.

— On treatment with tolbutamide 0.5 g. three times a day.

glucose, but the metabolism of the glucose within the cell differs. Thus diguanides appear to inhibit certain respiratory and oxidative enzymes, such as cytochrome oxidase. The glycogen content of liver and muscles falls, oxygen utilisation is depressed and lactic acid production rises. Hypoglycæmie drugs could also achieve their effects by decreasing the output of glucose from the liver as do the sulphonyl-They could inhibit the enzymes which degrade insulin in the body, as is the case with much larger doses of sulphonylureas than are used in clinical practice. They could also inhibit various physiological antagonists of insulin such as the steroid hormones from the adrenal cortex, or adrenaline from the adrenal medulla, or growth hormone and corticotrophin from the anterior pituitary, or glucagon from the a-cells of the pancreatic islets. Extirpation experiments in animals and use of the sulphonylureas in patients with various endocrine diseases have shown that the action of these compounds cannot be explained by neutralisation of the above hormones. Another possible method of maintaining a lowered blood sugar would be by inhibiting the absorption of food from the gut. This is clearly not the method of action of the sulphonylureas, since they do not affect the upward slope of the oral glucose tolerance curve even although they may lower all the blood sugar readings. This is

illustrated by Fig. 2, and Fig. 1

shows for comparison a normal

glucose tolerance curve. Relatively little work has been done on the mechanism of the hypoglycæmia achieved by the other compounds mentioned in the text.

#### Clinical applications

Here again the most suitable standard of reference in considering oral antidiabetic compounds is insulin, the efficacy and minor disadvantages of which are well established. The disadvantages include the danger of producing too low a blood sugar and thus disorders of behaviour or unconsciousness, and the occasional resistance to its action, although insulin in sufficiently high doses will not fail to lower the blood sugar. If a hypoglycæmic drug is to compete successfully with insulin it must be able both to increase the utilisation of glucose in the peripheral tissues and to decrease the output of glucose from the liver, thus correcting the abnormalities at these two sites in diabetes. These effects should be produced without the risk of toxic side reactions such as those produced in the nerves by guanidine over 40 years ago or to the liver by its derivative Synthalin in the late 1920's. Carbutamide, an anti-bacterial sulphonylurea has now been banned in the U.S.A. because of occasional toxic effects on the bone-marrow which interfere with the generation of blood cells.

The oral hypoglycemic compound which has received the widest use is another sulphonylurea, tolbutamide, although chlorpropamide may

later prove to be the more popular since it has a greater hypoglycæmic potency and longer duration of The majority of human diabetics have enough insulinproducing cells to respond to the hypoglycæmic action of the sulphonylureas, but many such diabetics are better and just as effectively treated by reducing their weight to the normal average or just below it. Probably some 20% of diabetics are suitable for treatment with the sulphonylureas, and it is fortunate that many of the older subjects with poor vision, who would find injections difficult, come into this category. The risk of producing excessive hypoglycæmia with these compounds is much less than that with insulin.

There remains the problem of oral treatment for completely insulin-deficient diabetics who usually develop the disease early in life. No oral treatment at present available can replace insulin in such cases, although some partial success has been obtained with the diguanide compounds, especially where the insulin requirement has been small or moderate. Although the mode of action of the diguanides appears to be unphysiological, patients receiving these compounds as their only treatment may feel fit unless they are troubled by the common early side-effects such as nausea and loss of appetite, or the later ones such as weakness and weight loss. Salicylates also exert a hypoglycæmic effect in insulindeficient diabetics, but relatively high doses have to be used and Even side-effects are common. where the diguanides or salicylates are inadequate as the sole treatment of a diabetic they may reduce the insulin requirement and stabilise the diabetic control.

#### Summary and conclusions

The chemistry, mode of action and clinical applications of the principal synthetic hypoglycæmic drugs have been discussed. Since in no case is the mode of action identical with that of the natural hormone, insulin, none of these compounds can automatically be used for the treatment of any given diabetic patient. A decision regarding which, if any, of these drugs is to be used must be made by the physician according to the features of the case and in the light of his own experience.

Chemists will no doubt persist

in the present empirical search for an oral substitute for insulin. The compounds discussed here, particularly Hypoglycin A, are capable of further development, and progress in the synthesis of polypeptides may shortly lead to the preparation of a substitute having a true insulinlike action.

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#### **Industry's Publications**

Know your peppermint. An attractive and informative leaflet on peppermint oils has been issued by Stafford Allen and Sons Ltd. It is the first of several publications planned for products of special interest to food manufacturers.

Steriliser-detergents. Useful formulation data on steriliser-detergents and detergent-sterilisers is given in technical service bulletin 68/1 from Monsanto Chemicals Ltd.

Stoker's manual. A new edition of this booklet has been issued (price 2s. 6d.) by N.I.F.E.S., 71 Grosvenor Street, London, W.I. The 12 chapters give the stoker complete information on the economical running of boilers and the many clear illustrations and diagrams help considerably to explain technical points.

Friction charts. Sturtevant Engineering Co. Ltd. have published a set of charts to meet the need of fan engineers and those concerned with ventilation, air conditioning, pneumatic conveying, etc., for data on friction of air in ducts and pipes. This valuable publication (No. 2007) is available from Sturtevant, price 7s. 6d.

Fibreboard packaging. The fourth issue of "Case," the journal of Reed Corrugated Cases Ltd., contains articles on various aspects of fibreboard packaging. Colour photographs illustrate the many attractive printing effects now obtainable with fibreboard

Photo chemicals. The first two British Standards in a series for photographic chemicals have just been issued. They deal with Metol (B.S. 3105: 1959) and Hydroquinone (B.S. 3103: 1959) respectively.

The new publications specify purity requirements for grades of material suitable for photographic use; and the object of the committee of experts which prepared them is, in due time, to produce a series which will cover the basic range of chemicals used in photo-

The standards may be obtained from the British Standards Institution, 2 Park Street, London, W.1. 4s. each

net.

### Glycols and Polyglycols

By Leon Raphael, M.SC., F.R.I.C.

#### 2. PROPERTIES

In the first article in this survey, Mr. Raphael discussed the chemistry and manufacture of glycols. Here he considers the properties of these chemicals which have led to their use in antifreezes, humectants, air sterilisers, cosmetics and pharmaceuticals. These applications will be reviewed in the third and final article.

THE glycols or diols are dihydric alcohols and as such are intermediate between the monohydric alcohols such as methyl or ethyl alcohol and trihydric alcohols such as glycerol. With the exception of the higher molecular weight polypropylene glycols, they are all water-soluble. The polypropylene glycols become water insoluble when the molecular weight exceeds about 900. The glycols are all colourless viscous liquids except 2-butyne 1.4 diol (see above) and the polyglycols of molecular weight greater than 400 which are colourless solids with fairly low melting-points. (See Tables I and 2 in the first article.)

#### Azeotropes

Like all monohydric alcohols, the glycols form azeotropes or constant boiling mixtures with several organic liquids. Although there appears to be no record of any industrial process utilising glycols for azeotropic distillation, it is possible that they could be utilised for recovery of solvents or the separation of liquids with very close boiling-points. Some investigations have been reported9 in which ethylene glycol was used to separate ethyl acetate from ethyl alcohol and to separate naphthalene from thianapthene. Styrene and o-xylene have boiling points differing by only 4°C. The azeotrope of ethylene glycol and o-xylene boils 7°C. lower than styrene and might therefore be useful in their separation. Some azeotropes of glycols are listed in Table 3.

#### Freezing-point depression

The freezing-points of pure glycols are often difficult to determine, because of their high viscosities at low temperatures. Of more interest, however, is the effect which glycols have in lowering the freezing-point of water. On a weight basis ethy-

Table 3. Some Azeotropes of Glycols

	Other component	B. pt. (°C.) of azeo- trope	% w/w glycol
Ethylene glycol	Chlorobenzene Nitrobenzene Aniline o-Xylene Naphthalene Menthol	130 186 180-5 139-6 183-9 188-5	5.6 59 24 16 51 51.5
Diethylene glycol	Nitrobenzene Ethyl salicylate Pyrocatechol Methyl naphthalene	210 225 259 225	10 30 46 39

lene glycol is the most effective of the glycols in this respect and for this reason is widely used as an antifreeze in car-radiators. This application will be discussed later. While other materials produce this same effect, glycol has been found to be the most successful anti-freeze. Brine solutions are very corrosive and methanol evaporates rapidly with an unpleasant and toxic odour. Methanol also has the disadvantage of a low flash-point while glycols up to quite a high concentration in water are non-inflammable. The eutectic composition of ethylene glycol and water, that is where the lowest freezing-point of the mixture occurs, has been found to be in the region of 60-80% glycol.

Diethylene glycol and triethylene glycol are solvents for nitrocellulose which is present in the paint-finishes on cars and for this reason is not recommended in antifreeze preparations. A comparison of the freezing point depressions of glycols in water is given in Table 4.

Hygroscopicity

Like glycerol, sorbitol and monohydric alcohols, glycols are very hygroscopic and the amount of water absorbed by a given weight of glycol depends on the relative humidity of the atmosphere. Table 5 shows the concentrations of the various glycols in water in equilibrium with the stated relative humidity. The polyglycols become less hygroscopic with increasing molecular weight (see Table 2).

Table 5. Humectant Values of Glycols given as % w/w of Glycol in Water at 60°F. in Equilibrium with the Given Relative Humidity

R.H. %	20	40	60	80
Ethylene	92.2	81-3	61-5	37-5
Diethylene	92.5	83-0	67-0	47-0
Triethylene	96-2	88-5	70-0	45-0
Tetraethylene	96-8	88-8	72.0	50-0
Propylene	93.7	84-0	72.0	52-0
Dipropylene	92.9	85-8	74-0	55-0
Glycerol	95-0	87-0	74-0	52-0

#### Solvent power

Glycols are miscible with lower aliphatic alcohols, aldehydes and ketones, but are insoluble in hydrocarbons. The lower aliphatic solvents act as coupling agents (mutual solvents) for glycols and hydrocarbons. Ethanolamines, phenols, and many inorganic salts are soluble Vinylite resins and in glycols. cellulose acetate are insoluble in most glycols. The solvent properties of propylene glycol are impor-tant as this is the only glycol accepted as completely non-toxic and can be used for foods and pharmaceutical preparations. It is

Table 4. Freezing-points (°C.) of Aqueous Solutions of Glycols

	1 au	C 4. 116	ering-hor	1102 ( 00) 0	u vdaco	as solution	10 01 01	1000
	% Glycol w/w	Ethylene glycol	Diethylene glycol	Triethylene glycol	Propylene glycol	Dipropylene glycol	Glycerol	Methanol
ı	10	-3.5	-2.5	-1-6	-3.2	-1.6	-1.6	-6-6
	15	-5.8	-4	-2.8	-5.2	-3.3	-3.2	-11.0
	20	-8	5	-4-4	-7.5	-4-4	-48	-15-4
	25	-11.5	-7	-6	-10	-6.7	-7.0	-20-8
	30	-15	-9	-8.3	-13	-8-9	-9-5	-26-4
	35	-19	-12	-11	-16-5	-11.7	-12	-32.6
	40	24	1.0	14	20.6	16	15.4	30 F

a solvent for vanillin, eucalyptol, menthol, methyl salicylate (oil phenobarbital, wintergreen), sodium iodobismuthate, sulphasulphadiazine, sulphapyridine, thiazole, thymol, as well as for vitamins A and D and a-ionone. Propylene glycol is also a solvent for several essential oils. Aqueous solutions of propylene glycol, while having a lower solubility of essential oils, are nevertheless found to be quite adequate in solvent power, so making the use of propylene glycol more economical.

Table 6. Solubility % by Weight at 25°C.

Conc. of propylene glycol
in water w/w

Solute	20%	60%	100%	
Cassia oil	0-21	0.85	00	
Clove oil	0-12	0.26	00	
Lemon oil	0-03	0-32	0.81	
Vanillin	2.09	12-6	20-2	

Chemical reactions of glycols

Esters of Organic Acids—Just as alcohols react with acids to form esters, so glycols form glycol esters.

Polyesters are formed by reaction with polybasic acids. Ethylene glycol condensed with terephthalic acid forms the linear polyester—*Terylene*. Phthalates and maleates are cured by polymerisation with another monomer. Diethyl oxalate reacts with ethylene glycol to form a cyclic dione.

The most important inorganic ester is the nitrate. Ethylene glycoldinitrate and diethylene glycol dinitrate are used as explosives.

The glycol ethers can be prepared by reaction of a glycol with dialkyl sulphates. However, these ethers are prepared industrially by

reaction of ethylene oxide and the corresponding alcohol. Dehydration of glycols yields dioxanes.

Aldehydes are also produced during this reaction. Aldehydes and ketones react with glycols in the presence of a catalyst to yield dioxolanes.

$$\begin{array}{c} \text{CH}_{2}\text{OH} \\ | \text{CH}_{2}\text{OH} \\ \text{CH}_{2}\text{OH} \end{array} + \text{RCHO} \longrightarrow \begin{array}{c} \text{CH}_{2}\text{--O} \\ | \text{CH}_{2}\text{--O} \\ \end{array} + \begin{array}{c} \text{R} \\ | \text{H}_{2}\text{OH} \\ \text{CH}_{2}\text{--O} \\ \end{array}$$

Dialdehydes form cyclic acetals.

Oxidation of ethylene glycol yields glyoxal as well as acetaldehyde, formaldehyde, glycolic acid and oxalic acid. These oxidation products, particularly the acids, are the cause of corrosion of metals by glycol solutions at elevated temperatures. This corrosive action can be inhibited as will be shown later.

When boiled at atmospheric pressure, the glycols tend to decompose slowly with eventual darkening of colour.

9. Chem. Abs., 1956, 50, 15154; 1954, 48, 11759.

#### Correction

We regret that Fig. 2 in the first part of this article (June p. 247) was incorrectly captioned. It illustrates the pressure hydrolysis of ethylene oxide to ethylene glycol.

#### **Emetine and Related Alkaloids**

The ninetieth meeting of the Fine Chemicals Group of the Society of Chemical Industry took place on May 15, following the annual general meeting of the Group.

Dr. H. T. Openshaw, head of chemical research at the Wellcome Research Laboratories, delivered a lecture entitled, "New Developments in the Chemistry of Emetine and Related Alkaloids." He pointed out that it was just about ten years since structures of the ipecacuanha alkaloids were finally established and that since then work on the synthesis of emetine and its stereochemistry has been satisfactorily concluded.

An observation by Sir Robert Robinson that emetine could be derived by a biogenetic process similar to that proposed by Woodward for strychnine enabled the structure of the ipecacuanha alkaloids to be linked with the large group of benzylisoquinoline alkaloids. One of the problems of the synthesis of emetine lies in the fact that there are four asymmetric centres in the molecule, but a valuable aid to the identification of synthetic products in this field lies in the reaction of emetine or its isomerides being easily oxidised to rubremetine, a red quaternary Dr. Openshaw decompound. scribed how he and his collaborators embarked on a synthesis without regard to the stereochemistry and was able to show that the final product did indeed give dlrubremetine, spectroscopically identical with rubremetine obtained from emetine itself.

The first claim to the synthesis of emetine was made by Evstigneeva, Preobrazhenski and co-workers in 1950, and although Dr. Openshaw was critical of the methods they had used for identifying some of their intermediates and final product, it has later been shown by van Tamelen that the method is reproducible although the separation of the isomers had to be modified. A related route was used by Barash and Osbond, who described in July 1957 a synthesis of emetine itself and several of its stereoisomers. In this work, the isomers are clearly differentiated by paper chromatography and by the use of infra-red spectroscopy for control purposes. The foregoing syntheses were undertaken in the absence of any detailed knowledge of the stereochemical configuration of emetine and were therefore rather "hit-or-miss."

Dr. Openshaw then dealt with the work carried out by Battersby, who by using Craig's countercurrent extraction methods isolated a new alkaloid, protoemetine, from the Ipecac alkaloids and the structure of this was such that the presence of Sir Robert Robinson's earlier suggested biogenetic intermediate was confirmed. Using this intermediate Battersby and his colleagues have been able to establish the configuration of natural emetine. Conformational arguments and correlation of structure with

the alkaloids of the indole series whose configuration has already been established, indicate the final structure shown, which was confirmed by a stereospecific synthesis.

Protoemetine

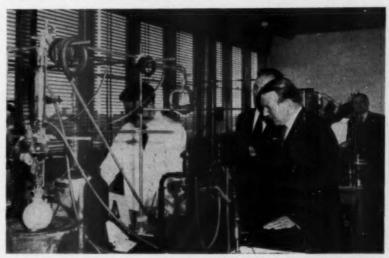
Emetine

The same conclusion was also reached by van Tamelen.

In the discussion which followed, Dr. Cohen asked whether the yield of rubremetine was identical for each isomeride and Dr. Openshaw replied that the yields were at least 50%, but that no direct evidence of differences were noted. Foster asked whether synthetic emetine was amorphous or crystalline since in his experience emetine had been a very difficult substance to obtain crystalline for many years, although on one occasion in his laboratory they had obtained one batch which was crystalline. The lecturer expressed regret that Dr. Battersby did not have any of Dr. Foster's seeds available to him at the time of his work. Dr. Osbond also commented that in his work at Roche Products they too had obtained an amorphous base which did, however, yield crystalline derivatives.

Dr. J. H. Wilkinson commenting on an earlier question by Dr. Walls regarding the effects of methylation in biochemical processes, pointed out that methylation in the thyroxine field confirmed the lecturer's observations that methylation usually blocks further biochemical reactions, and also pointed out that catechol amines are partially methylated in the body.

#### New Research Laboratories for Evans Medical



The Minister of Health, Mr. Derek Walker-Smith, inspecting one of the laboratories in Evans Medical's new research block at Speke.

COMPLETION of Evans Medical's new research laboratories at Speke, which were opened recently by the Minister of Health, Mr. Derek Walker-Smith, mark the culmination of a development programme which was initiated in 1941, when construction of new works at Speke was started, following the almost total destruction by enemy action of the company's head office and works in Hanover Street and Seal Street, Liverpool. Since then, the manufacturing activities of the company on Merseyside have been progressively rehoused in the new works at Speke, while part of the research laboratories of the company have been transferred to the new research laboratories.

Speaking at the luncheon which preceded the opening of the new building, Mr. I. V. L. Fergusson, chairman and managing director of the company, said that only approximately 20% of the 30-acre site had been developed. The buildings now completed, at a cost of £250,000, house general services designed to meet the full needs of the complete development and include a library, conference room and small animal breeding unit, as well as the essential engineering installations which are fed from the company's power house through a conduit constructed un-

derneath the road. The building also houses departments devoted to organic chemistry, microbiology, pharmacology, pharmaceutical formulation, and the analytical research and assay laboratories. There is also a suite of laboratories not yet allocated to a particular task and as yet to be equipped. Bacteriology, immunology, virology and biochemistry departments are still housed at the Evans Biological Institute at Runcorn adjacent to the factory making these products.

The new building, which was designed by Mr. V. Short, the company's architect, covers more than 35,000 sq. ft. and will essentially be concerned with investigations of a fundamental long-term character, with the scientific assessment of promising products which may emerge from this and with pharmaceutical formulation research. About 100 technologists and technicians work in them at present.

Pyrethrum formulation. A Formulators' Manual is being distributed to insecticide formulators by the African Pyrethrum Technical Information Centre Ltd. Translations of the text in French, German, Italian and Spanish are being printed. The need for such a manual was dictated by the rapidly increasing use of African pyrethrum throughout the world.

## Ethylene Oxide and Trichlorethylene from Acetylene

Automatic Process Control at German Factory

FROM an undertaking concerned exclusively with buna rubber and its by-products, the Chemische Werke Hüls A.G. of Marl, Western Germany, has within a few years developed into a company selling 600 different chemicals to all parts of the world. Turnover has increased from 815 million marks in 1958 to 528 million marks in 1957. While its main products are synthetic rubber and plastics, the company is also a large producer of ethylene oxide and trichlorethylene. Acetylene, produced from hydrocarbon gases by the electric are cracking process, is the basic raw material. Trichlorethylene, for instance, is derived from acetylene and chlorine, tetrachlorethane being obtained in the preliminary stage, which is then dissociated into trichlorethylene and hydrochloric acid. This plant is controlled from a central station on the wall of which a graphic panel is mounted; it reproduces in a very realistic manner the functions of the various apparatus and the flow of production. Since at the time this plant was built recording control instruments with comparatively small-size fronts were already on the market, use was made of them in the graphic panel, analogous to their effective position in the plant.

Automatic controllers ensure that temperatures, pressures, rates of flow and liquid levels are maintained constant.

The most modern automatically controlled plant at the Chemische Werke Hüls is that for the manufacture of ethylene oxide, which is obtained from ethylene and air by the contact process.

Ethylene oxide, which is a gas at normal temperature, has to be stored in a cooled condition or under pressure in its liquid form. It is much in demand in a number of industries. When processed to glycol it serves as an anti-freeze solution for car radiators. Another of its applications is as braking fluid for motor vehicles. The textile industry, the detergent industry, manufacturers of printing



Instrument panels and control desks in the ethylene oxide plant at Chemische Werke Hüls, W. Germany.

inks and pharmaceuticals, the paper industry and even pencil and rubber factories make use of ethylene oxide for the manufacture of their products.

The central control station of the ethylene oxide plant also has a graphic panel showing the flow of production. Control lamps are installed instead of control and measuring instruments. Altogether 35 different operations have to be automatically controlled. Once again there are pressures, temperatures and liquid levels which have to be maintained constant, and the plant has a total of 281 warning lights.

One hundred and eighty - six recording instruments and 229 indicating instruments are accommodated in the central control station, from which point the temperature can be measured at 250 different positions throughout the plant. Six electronic compensating recorders are located in a single control panel; within 48 sec. each of them records 12 different temperatures.

The automation of chemical

plants produces a number of advantages. Whereas in the older type plants operators had to go out in all weathers to read instruments and adjust valves, this is now done from a central control station, which is more like the navigating bridge of an ocean liner than what one generally expects to find in a chemical factory. A further advantage of these installations is that products which are either toxic or corrosive no longer need to be led through the control stations, since their conditions are transmitted to the central control unit through telecommunications equipment, either electrically or pneumatically. Additionally, production flows more smoothly and there is greater uniformity of the product. If one bears in mind that it took a man, working all out, from 4 to 6 min. to close a large slide valve, and that in the modern plant the same valve can be closed within 45 sec. by means of a regulating device, it will be appreciated that factors affecting the purity and quality of a product are much more easily controlled.

#### PROGRESS REPORTS

#### **Pest Control Chemicals**

By D. P. Hopkins, B.SC., F.R.I.C.

Insecticide granules • Sugar beet protection • Rotenone • Nematocides • Fungicides • Fluoroacetamide • Weedkiller in bar form

#### **Granulated insecticides**

AS almost all past work with insecticides has been with the object of developing very fine powders or liquid sprays in minute droplets, the idea of granulating insecticides hardly seems progressive. parently, granular size and shape offer advantages in some circumstances—e.g., when it is desirable for the applied substance to fall through a cover or barrier of vegetation without clinging to it. For several years granular insecticides have been considered in the U.S. and a recent review covers the subject most adequately.1 For mosquito control, granules get through overlaying vegetation and into the watery "living-space" of the pest; there the granules disintegrate and the toxic content is dispersed. In some crop applications, e.g., against corn borer, granules tend to lodge in branching parts of the plants, where the target pests often prefer to operate; at the same time dust does not cling to other plant surfaces, thus reducing the problem of residual toxicity. With soil treatment, e.g., against wireworm, the granular form of a suitable insecticide is not any more effective than the fine powder form, but dustless application is clean and convenient.

It is probably important to stress the fact that a granular insecticide is smaller than a granulated fertiliser. The size of granule aimed at has so far been slightly bolder than granulated sugar, slightly smaller than celery seed. Probably any insecticide can be produced in a granular form; well-known cases at present are aldrin, BHC, chlordane, cryolite, DDT, dieldrin, endrin, guthion, heptachlor, malathion,

rotenone, TDE, thimet and toxa-

Problems which have had to be faced deal with (a) the carrier, and a clay mineral, attapulgite, is much favoured; (b) the solvent, now up to 20% of the final product; (c) chemical stability; (d) avoiding dustiness; and (e) providing ease of granule breakdown under the action of Further work is clearly required in some of these directions, notably (a), (c) and (d). The formation of granules is a carrier problem, and in any fundamental sense the properties of the insecticide itself are almost unimportant. Granules formed with the carrier are impregnated with the insecticide, the latter usually being dissolved in a suitable solvent.

A second paper<sup>2</sup> has dealt more fully with granular insecticide manufacture. This indicates that manufacturers are tending to use rotary methods, together with drying—not markedly dissimilar from the wet-process method for granulating compound fertilisers.

(Readers who check the first of these two references should note that through a printing error the author's name was given incorrectly; the corrected name is stated in the reference list below.)

#### Sugar beet protection

The present U.K. use of systemic type insecticides to kill the aphid vectors of sugar beet virus yellows is strongly associated with a "warning system." Field men of the British Sugar Corporation check aphid populations in the various districts and issue warnings that enable growers to commence spraying, when needed, at the right time. In an area where the virus yellows

disease is common, the warning is given when there is one green aphid per four plants; in other areas, it is usually given when the aphid presence is one per plant. The operation of this system has been briefly described. Sugar beet is, of course, a somewhat unique crop, sold by contract to only one buyer; and sugar beet growing is localised. Similar pest control problems could not be as effectively approached for most other farm crops.

Weedkillers for sugar-beet acreages have also been discussed.4 and sodium trichloracetate or TCA as a specific herbicide for wild oats is clearly the most progressive recent development; propham or IPC may, however, compete in effectiveness with TCA when there has been wider experience of its use, and dalapon as a post-emergent wild oat control could become very useful if its slow action could be accelerated. For more general weed control endothal or the sodium salt of 8,6-endoxohexahydrophthalic acid seems promising. especially if mixed with propham. Six trials in 1958 showed a broad range of weed control for a 50-50 mixture of endothal and propham, particularly successful with broadleaved weeds.

This British report can be linked with a recent U.S. paper on endothal as a weedkiller for sugar beet.5 It must be a pre-emergent applied weedkiller for it can also be used as a crop defoliant. Trial data given show reduced weed populations and increased crop yields and sugar contents where endothal is used. One 1958 test showed that endothal application led to more than 3 extra tons per acre of beet as compared with hand weeding. Endothal does not persist in the soil for a long time; though this is advantageous in one sense, it is less so in anotherweed control throughout beet growth may not be achieved. Good distribution in the soil layer where most weed seeds are to be found is necessary; this can be achieved by special machines or by sprinkler irrigation application. In America as in Britain, interest in chemical weed control for the beet crop is rapidly increasing. Formerly, much weed control was carried out during the manual operation of thinning seedlings; now with this operation commonly mechanised, the weed problem is greater.

#### Rotenone

The sources of rotenone are the tropical plants derris and loncho-Harvesting their small carpus. roots and the transport costs arising from distant production areas leads to high prices. Other plant species have been studied for their contents of rotenoids at the Experiment Station in Puerto Rico.<sup>6</sup> Fourteen out of 16 species of Tephrosia were found to contain rotenoids in roots, seeds, or stems or leaves, some species containing rotenoids in all four "departments." These are leguminous plants. Their rotenoid content has been known for some but it had been (1937)time assumed that the amounts were too low for successful commercial extraction. Extraction techniques have advanced in two decades, and also rotenone from derris is probably no longer as cheap in relation to new insecticides. T. vogelii, one of the tested Tephrosia species, had from 0.65 to 4.25% of rotenoid content in its dry leaves. liminary trials indicate that this plant could be grown in the continental U.S. and that it would be adaptable to mechanised cultivation.

#### Nematocides

Another chemical approach to control of soil nematodes has been indicated.<sup>7</sup> Certain fatty acids—pelargonic, hepanoic, caprylic, caproic and undecylenic acids—at 1,000 p.p.m. as ammonium salts in water gave good control in contact with nematodes. But when passed through soils the solutions appeared to lose their nematocidal activity, though a formulation of pelargonic acid and hexadecylamine gave good control over tomato root-knot when used at 800 lb. an acre.

#### Fungicides

A Canadian paper<sup>8</sup> surprisingly reports equally successful control over wheat stem and leaf rusts with nabam, zineb and zinc sulphate. Four to five applications in the season were required for full control, and the first application must be early and made when only traces of rust were observable. The dosage rate for zinc sulphate was <sup>3</sup> lb. per acre. It has, of course,



Testing a new agricultural chemical made by Farbenfabriken Bayer A. G. in one of the glasshouses of their Biological Institute at Leverkusen. The atmospheric conditions of the glasshouses can be controlled to produce various climatic conditions. Bayer market certain crop protestion products in Great Britain through Baywood Chemicals formed in conjunction with Burt, Bouiton and Haywood Ltd.

a lower cost than more complex synthetic fungicidal preparations. A Tasmanian paper<sup>9</sup> still gives Bordeaux mixture an important role in control of blackcurrant leaf spot (Septoria). The best control treatment shown in a number of trials was based upon this long-known copper fungicide as the initial spray (green top stage); following this, thiram, captan or Bordeaux mixture again could be used as a second spray when the fruit was half grown.

An English paper<sup>10</sup> discusses potato blight control in the light of 1958's severe wet-season attacks. one of the "worst blight years in living memory." Here with attacks of epidemic character the warning system for preventive measures is no less important than for sugar beet virus yellows; there is not, however, as intimate a connection between growers and "warners." As this article states, B.B.C. announcements or local press notices from N.A.A.S. officers are all that can be given out, and the " rest is up to the farmer." Forecasting can only help to ensure that first sprayings are made in time to prevent or minimise attack. Again Bordeaux mixture is given veteran pride of place—"there is no doubt that thorough spraying with Bordeaux mixture gives the longest protection

under the conditions of a severe blight attack." The use of copper oxychloride or cuprous oxide sprays, at higher recommended rates, seems preferred (in this article) as a second spray following initial Bordeaux mixture spraying. Zineb is said to have done better than was expected during the severe 1958 season; and modern Dutch preference for zineb as the initial spray, followed afterwards by copperbased sprays, is mentioned.

#### Fluoroacetamide

In 1950 sodium fluoroacetate was found to be a very effective systemic insecticide against aphid species, but its use could not be developed owing to its high toxicity. Related compounds have been investigated and it is now reported that sodium fluoroacetamide is an active systemic insecticide; being less toxic and slower in its action, it may be much safer to use than fluoroacetate.11, 12, 13 Fluoroacetamide has already been shown to be sometimes superior in effectiveness to schradan. remains to be shown whether it can safely be used with edible crop plants, but for control of aphid attacks upon ornamental plants (e.g., roses) it would seem to be efficient. A solution strength of about 1 part in 10,000 is needed for

aphid control on roses; phytotoxicity is not apparent, however, at dilutions less than 1 part in 1,000. Fluoroacetamide's toxicity per seand here one is not referring to possible residues-should not cause development difficulties. It is already ranked as a Part 2 substance under the Agriculture (Poisonous Substances) Regulations, which means that when used agriculturally it requires the same precautions as those demanded for parathion or schradan.14

#### Weedkiller in bar form

A new method of applying selective weedkiller to lawns has been reported.15 Wax, impregnated with 2,4-D, is made into a bar weighing about 3 lb. which is dragged over the lawn by attached strings. Advantages claimed are that this method of application obviates spray drift risks, and that the film of weedkiller and wax is stable to rain should rain fall after the application. This latter point is important for in a summer such as that of 1958 in Britain a dry period long enough to make MCPA or 2,4-D fully effective seldom occurred. The 3 lb. bar is said to cover up to acre of turf, though in warmer weather the softer condition of the wax may tend to reduce slightly the area coverable. For the area involved a retail price for a 2,4-D impregnated block of just under 30s. is not too expensive. Little about this new weedkiller bar seems to have appeared in the technical or horticultural press; nor can one recall mention of the method in the U.S. press, usually quick to report novelties.

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#### **Analytical Chemistry**

By W. I. Stephen, PH.D., A.R.I.C.

Tranquillisers • Drugs • Pharmaceuticals • Antibiotics • Vitamins Alkaloids • Essential oils • Cosmetics

DURING recent years considerable developments have taken place in the field of tranquillising drugs and many types of tranquillisers are now manufactured. An important series of these drugs is derived from phenothiazine, and includes compounds such as promazine, chlorpromazine, perphena-zine, trifluopromazine. The adzine, trifluopromazine. ministration of these drugs to the mentally ill creates certain problems of a purely analytical nature. It is possible to detect breakdown products of these drugs by simple urine tests<sup>1</sup> only if appreciable doses of the drugs are given. But the introduction of more active tranquillisers such as Fentazine and Dartalan has led to a considerable reduction in the dosage so that the existing urine tests are no longer reliable. Although this is a particular example of clinical analysis and not really pertinent to this review, it does to some extent emphasise the need to have available sensitive methods for the analysis not only of the original drugs but also for their possible breakdown products, in this case, probably phenothiazone or a simple nuclear substituted derivative. Previous articles in this series2 have mentioned the analysis of some tranquillisers and the present review begins with an account of some recently described analytical procedures.

Tranquillising drugs

French workers3 have studied the chromatography and detection of a number of N-substituted phenothiazines. A descending technique on Whatman No. 1 paper is used, the solvent system being acetone-M-sodium acetate-M-acetic acid in the ratio 2:4:1 by volume. The spots are detected by exposure to iodine or bromine vapour, by the iodobismuthate reagent by fluorescence in U.V. light or by photography at 2587Å. Phenothiazine itself remains at the origin.

N-Alkyl-amino derivatives of phenothiazine have been determined by simple oxidimetric titra-The substances examined are chlorpromazine, diethazine, and promethazine, which in acid solution react with ceric sulphate or potassium bromate to form a red semiquinoidal free radical which on loss of a further electron, becomes colourless. The course of the titration can be followed visually or potentiometrically. These drugs can be determined in pharmaceutical preparations containing such additions as caffeine, amphetamine, codeine, barbiturates, lactose or

Chlorpromazine can be deter-mined colorimetrically by its reaction with phosphotungstic acid.5 The pink colour which appears is specific for this substance and Beer's law is valid for the coloured complex in the range 100-300 µg. of The method is chlorpromazine. suitable for the assay of tablets for injection. solutions Dimethylaminoacetylphenothiazine can be determined by amperometric titration.6 The finely powdered sample containing 100-200 mg. of the drug is suspended in 20 ml. of water containing 1 ml. of concentrated hydrochloric acid; the resulting solution is titrated amperometrically with 0.01 M-silicotungstic acid at a potential of -0.05 v. (vs. S.C.E.). The titration is a very slow one, but the error is less than -1.5%.

Drugs

A new colorimetric method for the determination of rutin7 makes use of uranyl acetate as reagent. The orange colour has a maximum extinction with a 1:1 ratio of rutin to reagent and enables concentrations in the p.p.m. range to be determined.

Phenylethylamine derivatives such as noradrenaline, adrenalone and related compounds can be detected on paper chromatograms by their coloured products with isatin.<sup>8</sup> This reaction is sensitive enough to detect 1  $\mu$ g. of the substance in a spot of 0.5 cm. diameter. Ephedrine and adrenaline do not

give this reaction.

New colour reactions for P.A.S. (p-aminosalicylic acid) described recently,9 are those with furfural and with xanthhydrol. In the for-mer, the addition of fresh 1% ethanolic solution of furfural to 3-5 mg. of P.A.S. results on boiling with a drop or two of concentrated hydrochloric acid in the formation of an orange-red colour with a maximum at 540 mµ. The reaction is not very selective; primary aromatic amines interfere. Similar treatment with xanthhydrol gives an orange colour which turns rapidly to a characteristic dark green (maxima at 480 and 654 mu). Phenazone and phenols interfere with this reaction. A colorimetric method for the determination of sulphonamides makes use of diazotisation and coupling with re-sorcinol. 10 The colour produced is then compared with those from a series of standards. The method is suitable for sulphanilamide, sulphacetamide, sulphaguanidine, sulphapyridine, sulphathiazole and sulphadimidine. A similar methodin is recommended for sulphathiourea, thymol being used to couple with the diazotised material. The useful procedures of non-aqueous titrimetry find application in the determination of sulphonamides.12 The present work concerns 12 common sulphonamides, p-aminobenzoic acid and its ethyl ester, and anthranilic acid. The general procedure is to dissolve the sample in sufficient glacial acetic acid and then to precipitate the base perchlorate by adding an excess of standard perchloric acid in glacial acetic acid, in the presence of benzene, carbon tetrachloride or chloroform. After 30 min. the excess acid is titrated with acetous sodium acetate in the presence of 1-naphthobenzein as the indicator.

#### **Pharmaceuticals**

Cation exchange can be used to separate caffeine from antipyrine.<sup>13</sup> In the procedure described, an aqueous solution of the two substances is passed into a cation exchanger in the iron (III) form. The antipyrine is quantitatively adsorbed, but the caffeine is completely eluted by washing the column

with hot water. The caffeine is then determined in the eluate by spectrophotometric means at  $271\,\mathrm{m}\mu$ . The same method has been used to determine caffeine in the presence of antipyrine and phenacetin<sup>14</sup> within  $\pm 2\%$  in tablets containing all three substances. This is as good as the results obtained using the conventional phosphomolybdate procedure.

Combinations of aspirin, phenacetin and caffeine together or with codeine, barbiturates or antihistamines are analysed by a separation procedure using multiple partition chromatography. The separations are effected by the use of different combinations of columns filled with celite and one of the following solutions: N-sodium bicarbonate, aqueous ammonia, 4N-sulphuric acid, N-tartaric acid, N-tripotassium phosphate. The eluting solvents are chloroform, acetic acid in chloroform or trimethylamine in chloroform. The individual components are determined spectro-

photometrically in the cluates.

#### **Antibiotics**

When chloramphenicol is hydrolysed with 25% hydrochloric acid the amide linkage is broken; 1amino - 1 - p - nitro - phenylpropane-1: 3-diol and dichloracetic acid are formed. The latter can be removed along with excess mineral acid by evaporating the solution to dryness. The resulting amine hydrochloride can be determined16 by the procedures of non-aqueous titrimetry. Visual or potentiometric indication can be used and mercuric acetate is added to mask the interference of the chloride ion. Acetous perchloric acid with crystal violet as indicator is used for the titration. The method can be used to determine chloramphenicol palmitate also; recoveries of 99.8-100.3% have been obtained.

#### Vitamins

Feigl and Jungreis<sup>17</sup> have described a spot test for the detection of vitamin B<sub>6</sub> (pyridoxine) in which the test sample is treated with 1-2 drops of phosphomolybdic acid and the mixture is warmed. The precipitate which forms is centrifuged off, washed with water, and then suspended in water. A drop of this suspension is placed on paper treated with Gibb's reagent (2: 6-dichloroquinone-4-chlorimine) and the paper is exposed to ammonia fumes. The yellow colour disappears and a blue stain results if 0·5μg, or

more of pyridoxine is present. An examination  $^{18}$  of the use of silicotungstic acid as a precipitating agent in the colorimetric determination of nicotinamide by the cyanogen bromide method shows that it helps to remove the interfering vitamin  $B_1$  and to stabilise the development of the colour. This is particularly evident in the analysis of multivitamin preparations and in deeply coloured vitamin B complex extracts containing iron and liver.

Hayden<sup>19</sup> has studied the effect of the sulphydryl group in the determination of thiamine with 6-aminothymol. Other substances particularly those of phenolic nature, give coloured products with this reagent, but of those examined in the present study, only cysteine gives a vellow colour in the same range of concentration as thiamine. This led to the assumption that the sulphydryl group was probably responsible for this reaction and in the determination of thiamine the thiazole ring is broken by alkali attack and the thiol liberated reacts with the 6-aminothymol. The present study indicates that the sulphydryl group is the main functional group in the formation of colour and the procedure can probably be applied to the determination of other similar compounds. Spectrophotometric studies of vitamins B<sub>1</sub>, B<sub>2</sub>, B<sub>6</sub> and nicotinamide alone and in mixtures have recently been made.20 By measurement of the absorption spectra of these substances in aqueous solution at pH values from 2-12, it is shown that the contribution of one vitamin to the total light absorption can be eliminated by measuring the extinctions of two solutions of the same concentration but at different pH values at a wavelength corresponding to the isobestic point. This enables complex mixtures to be analysed severally as two-component systems. The method is a rapid one and gives an error generally less than 2%.

A few methods have recently

A few methods have recently been described for the determination of ascorbic acid. In one of these methods<sup>21</sup> about 25-250  $\mu$ g. of ascorbic acid in a total volume of 10 ml. of water is treated with 3 ml. of a solution of thionine containing 0-05 g. per litre. This mixture is contained in a standard tube and it is irradiated by exposure to sunlight or at a distance of 25 cm. from a 75-watt lamp. The unknown



The analytical laboratory, at the new Warren Spring laboratory (see p. 278) one of the features of which are the fume cupboards ranged along the inner, corridor walls. Each cupboard has its own fume duct discharging at roof level.

is then compared visually with standards given simultaneous treatment. Dehydroascorbic acid can be determined by reduction with  $\rm H_2S$  and removal of sulphide ions with mercuric eyanide. A simple polarographic method<sup>22</sup> which can be applied to multivitamin preparations requires not less than 50 mg. of ascorbic acid. Polarography is conducted in a potassium dihydrogen phosphate buffer containing potassium citrate and potassium chloride, the important voltage range being 0·1-0·15 volts vs. S.C.E. Results are accurate to  $\pm 3\%$ .

A new quantitative procedure for the determination of vitamin C in fruit juices, particularly blackcurrant juice, is based on the paper chromatographic method of Strohecker and co - workers.24 ethanolic extract is prepared and two aliquots are taken. One is reduced with H<sub>2</sub>S and both are then subjected to ascending chromatography using as solvent nbutanol and water in the ratio 2:3 with the addition of 2% of oxalic acid and traces of potassium cyanide. The pH of the solution must be adjusted to 2.6-2.7 before chromatography and the spots are detected by spraying with ammonium molybdate. The aliquot which was first reduced contains the total ascorbic acid, and the total vitamin C and the ascorbic

acid are determined by Tillman's reagent, followed by back-titration with Mohr's salt.

#### Alkaloids

New procedures are constantly being developed for the determination of reserpine and related alkaloids. A titrimetric method25 suitable for determination of the total alkaloids in Rauwolfia involves preliminary extraction of the powdered root with a solvent mixture of chloroform, ether and ethanol (90%) in the ratio 8:23:2.5 and saturated with ammonia. extract is treated with 0.5 N-sulphuric acid, the acid layer is made alkaline with ammonia and the alkaloids re-extracted into chloroform. This solution is then titrated with 0.1 N-sulphuric acid using screened methyl red indicator. Another method26 applicable to pharmaceutical products makes use of the reaction of reserpine in 1:2dichloroethane as solvent with methyl orange. On the addition of ethanolic acid, a coloured complex (max. 528 m $\mu$ ) is formed, the extinction of which is proportional to the concentration of reserpine in the range 0.3-15  $\mu$ g. per ml. of solvent. A method<sup>27</sup> applied to preparations containing theophylline relies on the initial extraction of theophylline from a chloroform extract with dilute aqueous alkali.

The dry chloroform extract containing the reserpine is titrated with p-toluene sulphonic acid in chloroform in the presence of dimethyl yellow as indicator. An error of  $\pm 2.5\%$  is obtained.

A new procedure28 for determining alkaloids by non-aqueous titrimetry makes use of the hydrochloric acid complex of aluminium chloroisopropylate. This reacts as a monobasic acid in the neutralisation of various alkaloids. reagent is soluble in chloroform and provides a stable solution. It is best standardised against pure codeine sulphate with dimethyl yellow or ethyl orange in chloro-benzene as indicator. The present study relates to the determination of codeine, quinine, quinidine, and papaverine, the average error being within ±1.1%. A subsequent paper<sup>39</sup> deals with the preparation of the reagent and its use in the determination of atropine, strychnine, and amidopyrine. Chloroform and chlorobenzene in the proportions of 3:1 provide a more suitable solvent mixture if the reagent is to be stored for any time. A paper chromatographic method has been recommended30 as a means of separating strychnine and brucine. The mixed alkaloids are first oxidised with concentrated nitric acid and the mixture is made alkaline with sodium hydroxide. The pH is then adjusted to about 4.5 with acetic acid and an aliquot of the solution is subjected to descending chromatography with formamide-chloroform (1:1) as the stationary phase and butanolacetic acid-water (4:1:5) as the mobile phase. The paper is then dried and sprayed with potassium bismuth iodide which shows up the strychnine at Rp 0.76; the bromine appears as a red spot at R<sub>p</sub> 0·1, characteristic of o-bruciquinone. This procedure has been applied to the quantitative determination of strychnine in nux vomica. The tropane alkaloids are determined photochemically<sup>31</sup> using p-aminobenzaldehyde in concentrated sulphuric acid as reagent. Atropine, hyoscyamine, and hyoscine react to give an orange colour suitable for photometric measurement. A highly selective test for solamine has been described recently by Clarke.38 With not less than 1  $\mu$ g. of the alkaloid, a steel-blue colour is obtained using 1% solution of paraformaldehyde in 90% phosphoric acid. Of 300 alkaloids tested.

only a-chaconine and solamidine behaved similarly.

#### Essential oils

When rose oil is treated with 2:4 dinitrophenyl-hydrazine, the corresponding hydrazones are formed from the aldehydes which are A solution of these present. hydrazones is subjected to chromatography on paper impregnated with dimethylformamide, and decalin is used as the mobile phase.33 This enables the following aldehydes in rose oil to be identified:

> acetaldehyde, propionaldehyde, valeraldehyde, nonaldehyde, cinnamaldehyde, salicylaldehyde, phenylacetaldehyde and

Similar procedures have established34 that Bulgarian clary sage oil contains decaldehyde, valeraldehyde, butyraldehyde, propionaldehyde, acetaldehyde, furfuraldehyde and carvone.

The essential oil content of the flowers of Rosa damascena Mill has been determined<sup>35</sup> by a micromethod involving distillation and measurement of the oil. method is suitable for enabling a selection of the most satisfactory plants to be made.

A method for the analysis of mixtures of grapefruit oil in lemon oil depends on chromatographing mixtures of the two oils on chromatostrips coated with silicic acid. The two oils yield 7-geranyloxycoumarin and 5-geranyloxy-7-methoxycoumarin respectively which are recovered and cleaved with acid to produce the coumarins. The fluorescence of these are measured in U.V. light in a photofluorimeter. The results seem to indicate that grapefruit oil contains another fluorescing substance which migrates at the same rate as 7-geranyloxycoumarin during the chromatography.

#### Cosmetics

Hydrocarbons in cosmetic preparations37 are determined by passing a solution of the sample in low boiling petroleum ether through a column of alumina or silica gel. This retains esters and higher fatty acids and alcohols, but the hydrocarbons remain in solution and can be determined by drying and weighing after the solvent has been removed.

Partition chromatography is used37 to analyse lipsticks of either

the "castor oil" or "oleyl alcohol" type. A preliminary treatment with trichlorethylene and acetone separates out the lakes and fillers and the filtrate is evaporated to give the oil bases as a residue. This is dissolved in heptane, mixed with silanetreated celite, packed into a tube and eluted with 50% ethanol. This removes fluorescein dyes and polyethylene glycols. The column is then treated with 95% acetic acid which removes the castor oil and oleyl alcohol along with oil-soluble dyes. Hydrocarbons and waxes remain on the column and are eluted with chloroform. The various fractions are purified and weighed and the constituents of each are identified by infra-red spectrometry.

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Organic intermediates. The Midland Tar Distillers Ltd. have issued a leaflet describing intermediateslargely alkylphenols and pyridine derivatives-now available in development quantities. Many have not previously been available in Britain.

Pencillin and hair waving. Among new technical bulletins issued by Robinson Brothers Ltd. is one on n-ethyl piperidine, used in penicillin purification, and one on monoethanolamine thioglycollate which can be used in place of ammonium thioglycollate in the manufacture of hair waving lotions.

PVC plastics. More PVC is produced in Britain today than any other plastics material. At 100,000 tons p.a. PVC represents one-quarter of the total annual British plastics production (400,000 tons). In over 40 pages, and with the aid of more than 80 photographs the story of Geon PVC and its many uses is unfolded in a new booklet from British Geon Ltd. The growing importance of rigid PVC in chemical engineering is revealed in a special section devoted to the use of this material in the fabrication of all types of chemical plant and equipment and in the production of piping for water mains, gas lines and effluent disposal systems, where the corrosion resistance of PVC is an important advantage over other materials.

Fatty acids. Technical Publication No. 3-12-hydroxystearic acid from Price's (Bromborough) Ltd., deals primarily with the use of 12hydroxystearic acid (hydrogenated castor oil fatty acids) in the preparation of multi-purpose lithium based greases, but it is also of growing interest to manufacturers of alkyd resins. Technical bulletin sheet "Prifac" brand castor oil fatty acids" covers ricinoleic acid and castor oil fatty acids based on firsts and seconds castor oil. These products are of interest to manufacturers of ricinoleate esters, printing inks, alkyd resins, pharmaceuticals, disinfectants, greases, lubrieating and cutting oils, printing and ball point pen inks and metallic soaps. Ortho move Ortho Pharmaceutical Ltd., have moved to new premises at Saunderton, Righ Wycombe, Bucks., which accommodate laboratory, manufacturing and administrative activities.

Abbott's new factory Abbott Laboratories Ltd., of Baker Street, London, the pharmaceutical manufacturers, have acquired from the Admiralty a site on the Isla of Shappey between Queensborough and Shaerness for expansion of their manufacturing facilities. The site will be developed in phases. The work will be handled by C.A.S. (Industrial Developments) Ltd.

Irish company formed Evans Medical Ltd. have set up a subsidiary company to be known as "Evans Medical (Northern Ireland) Ltd., to samufacture, package and distribute pharmaceutical products. The company has built new promises of 9,000 sq.ft. floor space in Ravenhill Avenue, Belfast, and expects to employ approximately 70 workers, half of whom will be man.

Armour reorganisation. The pharmaceutical and industrial chemical divisions of Armour Chemical Industries Ltd., have been separated. A new company - Armour Pharmaceutical Co.Ltd. - has been formed with the following board: H.A.Hardt (U.S.A.), J.A.Gilbernau (U.S.A.), W.F.Ticchurst, S.Comway-Lee and D.Purdie.

Mr.Ticchurst, who joined Armour 31 years ago and has been general manager of Armour Laboratories, has been appointed acting-chairman of the new company.

Mr.Conway-Lee is the company secretary.

Wellcome acquire Cooper McDougall. The Wellcome Foundation Ltd. have bought all the Ordinary shares of Cooper McDougall Robertson Ltd. at a price of £2 a share ex the proposed final dividend of 10% (less income tax) and the 2½% capital distribution. The veterinary business of each company, though differing in nature and character, is in some respects complementary to that of the other. Both Burroughs Wellcome Co. and Cooper McDoughll Robertson Ltd. will continue to trade as separate companies and no changes in sales policy are envisaged.

Shell Chemicals roorgamised. In view of the growth of the Shell Chemical Co.Ltd. and the further expansion to be expected from the current substantial capital investment programme, certain of the company's operations have been integrated into three Divisions. Manager of the Industrial Chemicals Division will be Mr.P.J.March. This Division will deal with solvents, general chemicals, resins and detergent products. The agricultural organisation will remain unchanged as the Agricultural Division under Mr.H.G.Huckle and will continue to sell its products through its existing sales channels. The Plastics Division will assume responsibility for polyolofins, polystyrens, P.V.C. and synthetic rubber. The Manager will be Mr.V.N.Luke. Under Mr.A.D.Buckland-Nicks who becomes Regional sales controller, the existing sales divisions will be responsible for the field sales of the products of the industrial chemicals and plastics divisions.

Lime merger The Lime Division of I.C.I. will be marged into the Alkali Division on 1 January, 1960, and will be known as the Buxton Lime Works of that Division. The general policy regarding production and sales of lime and limestone will remain unchanged. Mr.G.W.Talbot, who is at present a Director (Visiting) of the Alkali Division, will become a full-time Director of that Division with responsibility for like affairs. He will continue to reside at Buxton. Mr.F.C.Covill and Mr.C.S.Hall will be appointed local Division Directors of the Alkali Division and will also remain at Buxton. The Lime Division Chairman, Mr.L.B.Ryder, will reach retiring age on December 31 and will then relinquish his post. The Lime Division employs over 1,100 people and is the descendant of the Buxton Lime Firms Co.Ltd., which was itself formed by an amalgamation of thirteen of the most important quarrying and limeburning companies round Buxton in 1891.

Armour Hose Chemicals formed Hess Products Ltd. and the Chemical Division of Armour Chemicals Industries Ltd., have been merged under the name of Armour Hess Camicals Ltd., of which half the share capital is controlled by Armour and Co. of Chicago, whilst the other half is controlled by United Kingdom interests, including a substantial proportion by Associated Chemical Co. Ltd.

8.G.I. in Glasgow For the first time in 24 years the Society of Chemical Industry held its annual meeting in Glasgow. The meeting opened officially on Tuesday, July 7 with a welcome by the lord Provost of Glasgow which was followed by the address of the President, Sir Robert Robinson, F.R.S., in which he gave a fascinating review of modern trends and aspirations in organic chemistry. On Wednesday, Prof. John Hawthorn of the Royal College of Science and Technology discussed the hazards of strontium-90 caused by fall-out from nuclear bomb tests. He was able to console his audience with evidence that biological mechanisms reduce the amount of strontium actually ingested with food. The Society's Medal was then presented to Dr. Francis H. Carr, a former chairman of British Drug Houses. Dr. Carr called his address "Autonomy and Automation in Chemical Industry". In it he argues for more support by industry for fundamental research because in spite of automation the chemical industry needed the autonomy of individual endeavour. Industry should give more money to the universities for fundamental research.

At the annual dinner on Wednesday evening, honorary membership of the Society was bestowed on Prof.Karl Ziegler of Germany, the inventor of low-pressure polythene, and upon Prof.D.Marotta, the secretary of the Italian Chemical Society and a leading figure in Anglo-Italian chemical exchanges. The new president of the Society is M.Ernest Solvay of Belgium.

#### PROPLE

Sir Alexander Fleck, who will be 70 in November, will retire from the chairmanship and the board of I.C.I. on February 29,1960. He will be succeeded as chairman by S.P.Chambers who is 55, has been with I.C.I. since 1947 and has been deputy chairman since July 1952.

Dr.P.J.Carner, formerly deputy laboratory manager at Thornton Research Centre, Cheshire, has been appointed Director of Research at Shell Chemical Co's Carrington Research Laboratory, near Manchester, in succession to <u>V.N.Luke</u> who has become head of the new Shell Chemical Plastics Division in London.

Sir Owen Wansbrough-Jones will join the board of Albright & Wilson Ltd. on October 1. Since 1953 he has been Chief Scientist of the Ministry of Supply. It was in that capacity that he opened the Corrosion Exhibition at Westminster last April.

D.C.M. Selt has been promoted director of sales and J.M. Kershaw director of development and market research of Monsanto Chemicals Ltd. The Company's two divisional development departments have been unified under Dr. J.A. Gardner, as controller of development. O.W. Mirray, who joined the company in 1932, has been appointed chief engineer in place of Mr. Kershaw.

K. Strehel is no longer connected with the Kolmar Organisation. Frederick Steinfels has been appointed chairman and Daniel Steinfels managing director.

Philip C. Schimmer, who for the past two years has been with Pharmaceutical Specialities (May & Baker) Ltd. in their export sales division, has joined the board of Brome & Schimmer Ltd. as technical and development director.

W.T. Loverrove, sales director of Lautier Fils Ltd. has retired after nearly 40 years service with the company. He joined the board in 1954 and will continue to advise the company. Paul F. Spencer succeeds him as sales manager.

Arthol Ltd. have appointed two additional directors, K.Strebel who has had wide experience in aromatics, perfumery and cosmetics, and A.H. Cope who will be mainly concerned with administration and finance.

### PLANT AND EQUIPMENT

#### MAUTOMATIC METERING PUMPS

The DCL "M" range of metering pumps produced by the Distillers Co. Ltd. has been adapted for automatic control. Developed primarily for the accurate metering of small quantities of fluids, the new range can now be operated with pH, temperature, flow and other controls. The automaticallycontrolled pumps are said to be responsive to pneumatic signals of from 8 to 15 p.s.i.g., adjustment of the plunger stroke being carried out by a small, compact bellows motor operating through a mechanism designed to reduce "hunting." Available with different plunger sizes giving a range of capacities from 0-750 c.c. hr. to 0-37.3 litres/hr., the pumps are suitable for working pressures up to a maximum of 2,400 p.s.i.g., the maximum pressure depending upon capacity. The output from each pump is proportional to the air pressure, the control gear being designed to give maximum capacity at either 3 or 15 p.s.i.g.

Pump heads are interchangeable, the head in each case being easily removed and replaced by one of different capacity. Power is provided by a h h.p. totally enclosed induction motor. The suction and delivery valves, both of which are duplicated, are of 12% chromium non-corrodible steel balls specially lapped to ensure high volumetric efficiency. Valve bodies, seats and the valves themselves can be removed by slackening one set screw, to facilitate inspection and maintenance. Hydraulically operated diaphragm heads are available for applications where it is desirable to avoid having glands in contact with

the liquid being pumped.

Designed for all types of plant and laboratory applications, the pumps are stated to be particularly suitable for chemical and antibiotic manufacturing processes, water treatment, catalyst injection and similar operations where accurately adjusted flows are needed. For special applications standard pumps and motors can be modified to suit individual requirements.

#### ▶BOTTLE RINSER AND DRYER

A small low-cost unit for cleaning new bottles, the *Apex Rotary Hydro* rinser and dryer, is available from R. Powley and Sons Ltd. The machine, which has a capacity of 1,440 bottles per hr., incorporates an efficient rinsing and drying system.

Bottles are placed into their holders by hand and move round the treatment chamber, receiving first internal and external jetting with water at 150°F, and then prolonged jettings with hot



**Automatic Metering Pump** 

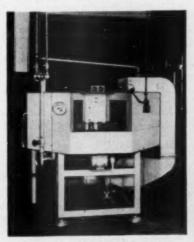
air before emerging dry at the unloading position. The manufacturers say the rinser-dryer can be made for all types and sizes of bottles and jars.

The machine requires 35 sq. ft. of floor space, and four adjustable feet enable it to overcome any slight irregularities on the floor surface.

### CONSTANT FEED RATE FOR CONTINUOUS BLENDING

The Avery 86N constant rate feed scale has been designed to give a constant rate of feed in blending processes and will operate satisfactorily, it is claimed, with most types of powered or granular materials providing they are free from large lumps or pieces.

Material is fed continuously to the



**Bottle Rinser and Dryer** 

scale into a feed hopper, from which it is withdrawn through an electronically self-adjusting gate opening by a moving belt, which then feeds the material to a moving weigh belt mounted on the scale. The scale is fitted with a "light and heavy" indicator mechanism incorporating an electronic switch which controls the size of the gate opening in the hopper, so that a constant weight of material is maintained on the weigh belt, this being shown by the indicator remaining at the "correct weight"

Both the feed and weigh belts are driven by synchronous motors, so that the belt speeds are constant. The belt speed is normally expressed in terms of "weight length per hour"; thus weight of material in lb. on the weigh-belt multiplied by the belt speed in weight-lengths per hour equals the weight of material in lb. delivered

The scale is made in two sizes, and on each size provision is made for changing the belt speed over a range of 200, 400, 600, 800 and 1,000 weight-lengths per hr., and also for changing the weight of material on the weigh belt. The smaller of the two machines has a chart graduated to 4 oz. light and heavy by ½ oz. divisions, enabling a very accurate control to be maintained up to a maximum capacity of 200 c. ft. per hr. The larger machine has a chart graduated to 1 lb. light and heavy by 1 oz. divisions and a capacity five times that of the small model.

Accuracy is said to be  $\pm \frac{1}{4}\%$ , depending on the nature of the material handled.

Each model is normally wired for 400/440 v. 3-phase 50-cycle electrical supply and is complete with relay cubicle and controls, contacts being provided which can be adapted to give an audible or visible alarm, or stop the scale automatically should the supply fail.

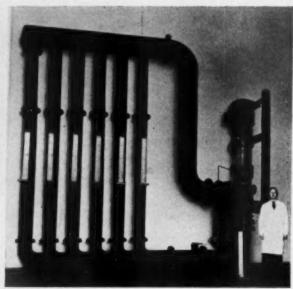
#### WATER DEIONISER

The new "Loughborough" water deioniser, model XD/2, incorporates a cartridge now coated in black nylon, which will give a substantial increase in its life by providing improved resistance both to chemical and mechanical wear.

The deioniser base is being fitted with a nonslip tripod stand to give greater stability where the unit is used on the bench, or in any unfixed position.

The unit is slightly dearer than the old model but there is no change in regeneration charges. Makers are the Loughborough Glass Co. Ltd.





Left: Special stillages for liquid gelatin in capsule factory. Right: Pump testing rig.

### MATERIALS HANDLING IN A CAPSULE FACTORY

Ninety per cent of the world's flexible gelatin capsules are produced by the R. P. Scherer Organisation for the pharmaceutical, veterinary and other industries, and materials handling is an integral part of the company's production processes. Methods used include roller and vacuum conveyors, hoists and trucks, and pallet and stillage systems.

Gelatin arrives at Scherer's Slough plant as a fine crystalline powder. It is tested for purity and viscosity, mixed with water and glycerin and then melted under vacuum. The viscous mass is transferred from the melter to stainless steel tanks of 500 lb. capacity, which are designed with sufficient under-clearance for use with Yale hand lift trucks.

Containers of different sizes and shapes carry the products for encapsulation. All are placed on flat wooden pallets which are stored in special racks. In conjunction with a Yale fork lift truck, this system is said to have relieved floor congestion and trebled storage space. Goods selected from storage are transferred from pallets to skids for movement by Yale hand lift trucks to the weighing department, where they are check-weighed into bins. A Yale electric hoist moves feeder drums over the scales.

Formulæ involving large numbers of ingredients are compounded homogeneously, as each aliquot must have the correct ratio of contents. The mixtures are poured into 45 gal. drums on stillages which incorporate heating and revolving units to prevent separation of drum contents between processes. The stillages were designed for use with Yale hand lift trucks.

Drums of the capsule fillers, still revolving on the special stillages, are trucked into position near the encapsulation machines by hand truck, and liquid is tapped from the drums as required. Tanks of gelatin are fetched from temporary storage by a Yale fork lift truck which lifts them on to the machines to serve as gelatin hoppers.

Two conveyor lines in the packing department deal with small and bulk orders. Capsules are normally bulk-packed in polythene bags and then fitted into cartons which are placed on stillages and moved by Yale trucks to the despatch bay.

Yale handling equipment used by the company comprises *Blue Streak* hand trucks, fork lift *Worksavers* and *Wizard* electric hoists.

#### TIN SEALER

A fully automatic tin sealing machine, said to be capable of sealing all types of multi-angle tins with a wide range of pressure-sensitive tapes, is available from John Gosheron and Co. Ltd. The taping heads revolve around the tin, which is stationary, and feed-in is by endless belt with an indexing mechanism which controls the flow of tins, ensuring that they are presented to the taping heads in sequence. Two heads each apply tape round one half of the tin, forming two overlaps.

Heavily rimmed lids are said to present no problem and the machine will apply cellulose, vinyl, polythene and paper self-adhesive tapes, it is claimed. A small compressor is incorporated to operate the pneumatic valves on the taping heads and speeds of up to 15 tins per min. are claimed.

#### PUMP TESTING RIG

In order to test the design of their new series of vertical axial flow propeller type pumps—the H Type-Kestner Evaporator and Engineering Co. Ltd. have erected a special rig. This embodies six vertical pipe sections each fitted with orifice plates with full diameter and half diameter tappings and mercury filled U-tubes. The pipes are arranged so that any number can be cut out for the testing of smaller pumps. Furthermore a valve is fitted into the horizontal bottom section in order to permit adjustment of the flow rate when testing the smaller pumps. It is obvious that, with the smaller total flow, readings across the orifice plates would be too low to provide significant figures, especially with all six pipes in operation. Pitot tubes are provided in both inlet and outlet pipes: two in each pipe at right angles to each other and in the same plane.

All pitot tubes are capable of traversing the pipe section.

It is necessary to fill the whole system with water prior to undertaking tests, and for this purpose the company installed one of their *J Type* pumps mounted on a small supply tank.

Once the system has been filled this pump is left running in order to make up for the liquor lost through the *H* pump (on test) overflow.

Tests have been made in the normal way measuring quantity in accordance with British Standard 1042:1943, and measuring head to British Standard 599:1945.

Power readings were measured by a watt meter reading Kw/2 phases. The rig is capable of handling liquor flows of up to 10,000 g.p.h.

#### **▶ULTRASONIC HOMOGENISER**

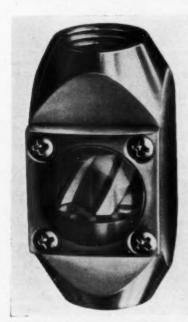
For many years there has been a need for a small versatile homogeniser at a reasonable price. There are many instances where the task involved does not justify an expenditure of over £150. Much of the equipment which has up to now been available has proved to be very limited in its range of application. The gap is filled by the new *Minisonic*. It is virtually three machines in one:

 A laboratory homogeniser with which small quantities of emulsions and dispersions can be quickly made. Up to 3 litres can be treated at one filling without external premixing.

(2) A small batch homogeniser for small batches of emulsions as required in the cosmetic industry or in hospital dispensaries.

(3) A pilot plant homogeniser with which an emulsification process can be worked out on a small scale and immediately transferred to production using the Rapisonic homogeniser.

The principle upon which the machine works is that of the liquid whistle covered by British Patent No. 695491 (and many foreign patents). It has been established as an efficient method of utilising cavitational forces in liquids and is described in many standard textbooks. A simple explanation is that a thin blade is made to vibrate in a liquid stream. These vibrations have a frequency of 18-22 Kcs, are inaudible to the human ear and of such a high level of energy as to induce cavitation. Intimate mixing is thus brought about



Telicator visual flow indicator.

and it results in truly homogeneous emulsions of very fine particle size.

During development work on the machine no limitations with regard to hot liquids were met. A novel feature enables a very wide range of viscosities to be processed with equal efficiency. All parts in contact with the liquid to be sonified are made in 18/8 stainless steel and corrosion-resistant materials. All surfaces are polished, which greatly assists in cleaning; if necessary the whole unit can be quickly dismantled. Built-in glass vessels and a three-way finger-tip control valve make for ease of operation and the Minisonic is supplied ready for plugging into a 15 amp. three-pin socket.



Glass tube cutter introduced by Electrothermal Engineering will cut tube up to 3 in. dia.

#### VISUAL FLOW INDICATOR

What is claimed to be an inexpensive and more compact version of the *Telicator* liquid flow indicator has been added to the range available from Sir W. H. Bailey and Co. Ltd. agents and distributors for the manufacturers, Dukes and Briggs Engineering Co. Ltd.

The unit is designed for insertion in pipelines, the ends being threaded \(\frac{1}{2}\) in. or \(\frac{1}{2}\) in. B.S.P.T., and for use with liquids having temperatures up to 80°C. It can be used with high or low pressure systems and with liquids of almost any viscosity, it is claimed. One of the main advantages over previous models is that it can be dismantled for cleaning without

breaking the pipe connections.

The new unit—known as K-type—
is 2½ in. long and contains a *Perspex*cylinder housing a rotor which is
located by pivots and sprung bearings,
so that the flow of a liquid is revealed
by the revolving rotor. The method of
pivoting the rotor is claimed to reduce
friction to a minimum, so that the rate
of flow of a liquid is hardly affected.



Minisonic model 3 laboratory and small batch ultrasonic homogeniser.

#### ▶GLASS TUBE CUTTER

Hard or soft glass tube up to 3 in. in diameter can be cut by a new instrument from Electrothermal Engineering Ltd. The tube is rotated against the cutting wheel and the resulting scratch mark is then held against a hot wire, localised heating causing the tube to part neatly. An adjustable backstop makes sure that the scratch is cut accurately around the circumference of the tube.

#### VIBRATORY MIXERS

A new range of vibratory mixers with no rotating parts and said not to require mercury seals or stuffing boxes was recently demonstrated at the Fenton works of QVF Ltd., industrial engineers in glass.

The mixers consist of a vibration generator, operating on alternating current, which vibrates the shaft at the same frequency, moving it up and down, in short strokes. A two-way switch is incorporated to alter the stroke length of the shaft, by means of which the mixing action can be increased or decreased as desired.

Available in three sizes, the mixers have capacities of from 10 to 3,000 litres. It is claimed that the extremely rapid vertical agitation of the horizontal blades quickly achieves perfect mixing by the rapid acceleration of chemical reactions. Shaft and plates are of stainless steel and the vibrator is totally enclosed. Special sealing assemblies are designed for specific applications.

The Vibro-Mixer was designed in Switzerland by Dr. H. Muller and is distributed throughout Great Britain by QVF Ltd.

For more information about the plant and equipment described please use the coupon on page 302

### New Products

#### Acne treatment

The German firm of C. H. Boehringer Sohn, Ingelheim, has developed a new treatment for acne vulgaris and other types of seborrhœic dermatitis. Clinical trials with more than 1,000 patients showed it to be superior to preparations containing mainly sulphur, salicylic acid, resorcin and other antibacterial substances. The new preparation is called *Thiōcarbin* and it is incorporated in lotions, tinctures and powders, in strengths of up to 0.55%.

### Demulsifying agent prevents oil pollution at sea

A new demulsifying agent which may make a significant contribution to the prevention of oil pollution of the sea has been developed by research scientists of D.S.I.R. and Glovers (Chemicals) Ltd. of Leeds. The material is a surface-active agent called *Fomescol*. It was originally designed to overcome certain boiler troubles in ships, for which it has proved successful.

Trials have now shown two other very important uses. The first is for tank cleaning in dockyards; the second is for the cleaning of tanker washings at sea, the disposal of which can be a major source of pollution. With the use of this agent, dry oil is recovered which can be used or sold. The great advantage of the agent, which is far superior to any similar agent available in the United Kingdom or abroad, is that more than 80% can be recovered for re-use.

The furnace fuel oil burnt in marine boilers is liable to form stable emulsions when contaminated with sea water. Such emulsions cause a number of troubles; chief of these are corrosion of tubes and slagging of refractories, making frequent re-tubing and relining of furnaces necessary.

The Fuel Research Station, D.S.I.R., was asked by the Admiralty to investigate the problem. All possible methods of demulsification were examined at the Station, such as addition of salts, use of steam, electrical techniques and filtration. The addition of surface-active agents was found to be the most promising.

Every agent available on the British market and numerous agents from foreign sources were examined, but even the best would not break emulsions with all oils. It was decided that the only thing to do was to synthesise a molecule specially for this

Facilities for such synthesis were not available at the Station. Three chemical manufacturers were approached and of these Glovers (Chemicals) Ltd. agreed to co-operate in developing an agent. A study was made of the effects of systematic

variations of the molecular structure of certain compounds on their efficiency as emulsion breakers and in this way an efficient molecular structure was brought to light and the agent has now been patented jointly under the name of Fomescol by D.S.I.R. and Glovers.

Fomescol shows no specificity in action with any oil so far examined, nor is any critical concentration shown. About 0-01 part of the agent dissolved in water is mixed with 100 parts of oil. The oil is then heated to 140°F, for 12-24 hr., when the water separates to the bottom with a clean interface. These particular boiler troubles have thus been solved.

Further trials were undertaken by the Admiralty on tank cleaning in dockyards. Again the results were satisfactory, the oil being recovered and more than 80% of the agent also being recovered for re-use. This is the first time that such a claim has appeared for any surface-active agent on the world market.

The tanks of tankers are frequently cleaned at sea, the normal method being to wash them with salt water, the resulting mixture of oil and salt water being collected and stored in one tank. If this mixture cannot be discharged ashore it must be pumped out into the sea. With Fomescol the oil can be recovered dry and sold, so that dumping of the residues will no longer be necessary. The cost is low, due mainly to the repeated re-use of the recovered solution.

#### Helmox reformulated

Imperial Chemical Industries Ltd., Pharmaceuticals Division's new formulation of Helmox is now available for sale to farmers. This new formulation contains 33% cyanacethydrazide in a phenothiazine base and is designed for the prophylaxis and treatment of lungworm infestation in farm animals. Literature on the new formulation is available and it should be noted that the dosage recommendations are different from those given in the original preparation. Distributors are asked to return any stocks of the old formulation or arrange for replacements when the company's representative next visits them.

Prices are: trial pack, 15s. retail, 10s. trade; "A" pack, 45s. retail, 30s. trade; and "B" pack, 260s. retail, 173s. 4d. trade.

The trial pack contains sufficient for one dose for approximately 7 calves, or 16 sheep or goats, or 32 lambs. "A" pack is approximately sufficient for one dose for 10 cattle, or 25 calves, or 56 sheep or 112 lambs. "B" pack is approximately sufficient for one dose for 66 cattle, or 166 calves or 372 sheep or goats or 745 lambs.

#### Anti-nausea drug

Burroughs Wellcome and Co. have introduced Valoid injection of cyclizine lactate for the prophylaxis and treatment of post-operative nausea and vomiting, drug-induced nausea, vertigo following fenestration operations, nausea and vomiting of pregnancy and other types of intractable vomiting when oral therapy is impracticable. "Valoid" injection is available in ampoules of 50 mgm. in 1 c.c., packed in boxes of 5, at a price of 6s. per box, subject to the usual discount.

#### Amphotericin B

The Squibb International Division of Olin Mathieson Chemical Corporation has developed a new antibiotic called Fungizone (generic term: amphotericin B), which is said to be effective in the following systemic fungus diseases, several of which have, until the discovery of this preparation, been fatal: cryptococcosis (torulosis); histoplasmosis; coccidioidomycosis; South American and North American blastomycosis; aspergillosis; South American leishmaniasis; and disseminated moniliasis. Several physicians it is stated, have also obtained beneficial results in using Fungizone for the treatment of cryptococcal meningitis.

Fungizone or amphotericin B is derived from a previously undescribed species of Streptomyces which was isolated at the Squibb Institute for Medical Research in New Brunswick (U.S.A.) from soil samples obtained from the banks of the Orinoco river in Venezuela.

Fungizone is generally administered by intravenous infusion; other parenteral routes may also be used. It is available as a sterile powder, packaged in vials containing 50 mg. of amphotericin B activity, for reconstitution in dextrose solution. According to the manufacturer, saline solution should not be used.

E. R. Squibb and Sons, Liverpool, are distributors in the UK.

#### Sheep dip

A sheep dip designed to meet requirements of farmers who prefer liquid to powder "dips" has been introduced by Boots Pure Drug Co.

It is liquid 400 Dip, concentrated for use one part dip to 400 parts water, and is the approved single dipping type containing aldrin and gamma B.H.C.

Boots have also introduced a new sheep marking fluid. It is claimed to persist under all weather conditions, yet wash away easily when the fleece is being prepared by the woollen manufacturers.

It is supplied in black, red and blue.

#### Clover control chemical

Boots have introduced Lornox Plus weedkiller, which does all that Lornox does and kills white clover, it is claimed.

It is the first product of its kind, and has an entirely different formula to the original *Lornox*, as it is partly based on the chemical CMPP introduced by Boots three years ago.

A 4 oz. bottle, which costs 5s., is sufficient to treat 80 sq. yds. of lawn.

#### Padiatric Tedral

William R. Warner and Co. Ltd. have introduced Tedral pædiatric suspension, a flavoured liquid preparation providing the equivalent of half a Tedral tablet in each teaspoonful. Although designed primarily for the child asthmatic, the manufacturers feel this presentation will also prove acceptable to patients who dislike, or find difficulty in taking, tablets. It is available in 4 oz. bottles at a trade price of 48s. per doz.

#### "Coomassie" blue

Imperial Chemical Industries Ltd., Pharmaceuticals Division, have developed a new product Coomassie Blue (Medical). It has been produced at the request of cardiologists for a reliable dye for the investigation of cardiovascular function. It is the sodium salt of anilino-hydroxyazonaphthalene sulphonic acid. The advantages of Coomassie Blue are that it is of low toxicity, it is well excreted and does not stain the patient's skin blue; large doses can be given without the need to control oxygenation of the blood.

#### Anthelmintic for husk

Co-operation between the Ministry of Agriculture and the Wellcome Foundation Ltd. has resulted in the production of a new treatment for "husk" in cattle, which is estimated to cost the British Agricultural Industry losses amounting to some £3 million annually.

The new product (diethylcarbamazine citrate) has been named Franccide and is now available to farmers through the veterinary profession. It is administered by intramuscular injection in the hindquarters of the animal.

"Franocide" acts in a different way to existing methods of controlling the lungworm which causes "husk." It attacks the larvæ of the lungworm as well as the adult worm, whereas other drugs only attack the worm after it has passed through the lungs of the animal and consequently after the damage has been done by its passage, in its immature form, in the lungs. In addition, Franocide has no side effects. It is issued in a form ready for immediate administration, thus eliminating any previous preparation by the veterinary surgeon. The complete course of treatment is three injections on successive days.

### **Packaging**

#### Double coated tape dispenser

The latest addition to the Minnesota Mining and Manufacturing Co. Ltd. range of taping equipment is the ME 71 double coated tape dispenser. Claimed to be easy to load and operate, it peels off the protective liner from Scotch brand double coated tapes, exposing the second adhesive surface, while the tape is being dispensed.

The dispenser will handle Scotch double coated tapes Nos. 400, 140 and

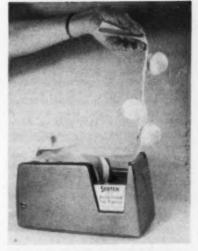
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#### New pack for Oxydent

Oxydent denture powder is now sold in Poly-Tainers made by the Plastics Group of the Metal Box Co. Ltd.

The flexible bottles, which are produced in two sizes, were designed specially for the job. Made of light blue polythene and fitted with a dark blue screw cap, they make pleasing packs which have the additional advantages of being light in weight and unbreakable.

The new pack makes use of both printing and embossing. The brand name is printed in white on the front of the bottle and directions for use are clearly embossed on the back.



The ME71 double-coated tape dispenser.

bottle are said to be virtually unspillable by accident. Hence this simple form of squeeze dispensing, which does away with the need for extra components, is suitable for a wide variety of liquid products which do not require an air-tight reclosure.



#### Detergent dispenser

Mil, the Co-operative Wholesale Society's liquid detergent for washing up, is also marketed in a Poly-Tainer flexible polythene bottle. The white polythene is printed in red, green and black, and the red screw cap, made of urea, is designed so that it can be pierced through the top and the detergent squeezed out efficiently and economically.

Though the cap, once pierced, cannot be resealed, the contents of the



#### Polystyrene capped corks

The Viscose Development Co. has introduced a plastic-headed bottle stopper. The head, made of polystyrene, is inert and is impervious to water and spirits. It is also claimed that it cannot swell or shrink and is odourless. An adhesive, specially formulated for the stopper, is said to guarantee that the head cannot be separated from the cork stopper unless the cork is split or broken.

Product names and company trade marks can be effectively produced on the polystyrene head. Tests already made by the company prove that the embossing technique produces a clearer and cleaner-cut outline, and intricate trade-marks lose none of their details. The trade mark or product name is readily identifiable by the purchaser and cannot be erased by handling.

The polystyrene head is available in many colours and the embossing can be coloured gold or in contrasting ink colours. Tooling for the engraving is approximately £25. Thereafter, the price of the stopper is stated to be competitive with wood and metal topped stoppers.

The stoppers can be sealed with Viskrings which shrink to fit the outline of the bottle neck. These can be printed in two or more colours with brand names or trade marks.



Poly-Tainer polythene bottles for liquid detergent and denture powder.

#### " Push-button " toothpaste

D. and W. Gibbs are now marketing

S.R. Toothpaste in a pressurised pack.
The new "push-button" pack, in
the red, white and blue S.R. colours, retails at 6s. 9d.



#### Pack for polio vaccine ampoules

Pfizer Ltd. now pack their 1 ml. ampoules of poliomyelitis vaccine in plastic containers. Each container is a six-ampoule pack consisting of two parts—a dark blue base made of polythene and a light blue cover of moulded high impact polystyrene with a gloss finish.

Polythene is used for the base because it is strong and pliable. Ampoule anchorage in each of the six separate compartments is by an arrangement of four fins, the resilient properties of which ensure good suspension. The surface of the outer sides of the base has fourteen ridges which grip the cover giving a firm closure.

The pack, the design of which permits the ampoules to be loaded by machine, is made by Universal Metal Products Ltd.



### Versatile corrugated packing

If strawpaper or a similar material is fed from the reel into a special machine with profiled rolls under the action of pressure and heat, a characteristic double wave is permanently imparted to it. The resulting resilient material is known as Carbion. If the feed of the material through the patterning rolls is inhibited, the double-wave pattern is telescoped into ridges. The density of the pattern is said to give a very high load-bearing capacity. It also becomes elastic and can be stretched, tending to contract again when released. This material is known as "flexible" Carbion, while the other is called "rigid", but it is rigid only insofar as it cannot be stretched.

When it is fully stretched, the flexible material with its ability to support heavy loads looks and behaves exactly like the rigid product, which is





(Top) A Carbion sleeve, scored top and bottom, folds round a tin. A tube of kraft paper, sealed at both ends with a gummed patch, completes the pack. (Bottom) Rigid Carbion in narrow strip is snaked between bottles, while sheets of the material act as liners between All glass-to-glass contact is layers. eliminated.

designed to give a cushioning effect. By partly stretching the flexible material, a combination of both properties is achieved, their balance depending on the degree of stretch. This feature is exploited by forming the flexible material into tubes or sleeves and by pulling them over objects requiring protection.

A typical example of the use of sleeves is shown in the top photo; it is a special diet sold by mail order in a cylindrical tin, which used to be wrapped by hand. The job is now done in half the time by inserting the tin in a sleeve, both ends of which are scored by the suppliers. The ends fold over

to protect the ends of the tin. The sleeved tin is inserted into a kraft paper tube, ready cut to the required To facilitate insertion, the tube is slightly larger than necessary, the "slack" being taken up by a fold made by the operator. The ends are folded over and secured with a circular gummed label and the address label is stuck over the fold in the tube to prevent it from catching against other items of mail.

It is stated that by performing these operations on a production-line basis, the turn-round of postal orders received has been considerably speeded up, and loss of coasignments due to the wrapper bursting open has been eliminated.

An application of the rigid material is shown in the bottom photo. A narrow strip of rigid Carbion is "snaked" between the bottles as they are put into the carton, while sheets of the same material are placed under. above and between layers of bottles. By this means, all glass-to-glass contact is eliminated, and since this is the most frequent cause of breakage, losses due to damage have been cut to a fraction of the former figure.

Made of decorative materials or laminated with paper which can be coloured or over-printed, carbion can make an attractive pack. It can also be laminated to many different kinds of special papers and foil, so that the finished product can be made pHneutral, moisture-repellant, greaseproof, soft or tough, transparent or coloured. In fact, Spicers, Ltd. who make it, claim that it can be adapted to fill almost any need.



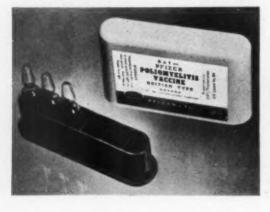
#### Sealing cartons

A tape applying machine which will seal cartons, apply tape to certain "special offer" packs and to small "special offer" packs and to small bundles of goods, is available from John Gosheron and Co. Ltd.

Known as the Spartan, it applies a shallow "U" seal of pressure-sensitive tape to the underside of a carton.

Operation is said to be extremely simple. The carton is brought in contact with the taping head and is then pushed along the platform which commences the underside seal. As the end of the carton passes over the mechanism a spring-loaded knife cuts the tape. Finally, a roller wipes down the tape at the rear end of the pack to complete the seal. According to the manufacturers any size pack can be sealed at both the top and bottom at speeds of up to 35 cartons a minute. The sealer can be set in the middle of a conveyor belt, used in the inverted position for top sealing and sideways for specialised tape applications. There are few moving parts, and the mechanism requires no maintenance beyond

The machine is avilable in two sizes.



This six-ampoule pack is used by Pfizer Ltd., for their poliomyletis vaccine. Each 1 ml. ampoule is held by an arrangement of four fins.

### NEW COMPANIES

These particulars of new companies have been extracted from the daily register of Jordan and Sons Ltd., company registration agents, Chancery Lane, London, W.C. 2.

J. F. Chemicals Ltd. 5.2.59. £100. Dirs.: not named. Sub.: A. D. M. Phillips, 2 Bedford Road, London, W.C.2

H. E. Niblett Ltd. 5.2.59. 103 St. Marks Road, Eastville, Bristol 5. To take over bus. of retail and dispensing pharmacy ed. on at Bristol by Harley E. Niblett. £5,000. Dir.: H. E. Niblett.

Castle Laboratories Ltd. 4.12.58.
77 Castle St., Hinckley. Mnfg. and dispensing chemists, etc. £100. Dis S. Woodward, S. West and S. Swart. Dirs.:

Cecil Norman (Cheltenham) 11.12.58. 15 Montrelies technology 15 Montpelier Walk, Cheltenham. Chemists, druggists, drysalters, wine and spirit merchants. £1,500. Dirs.: C. and T. R. I. Norman. £1.500.

R. H. Ferris Ltd. 12.12.58. 450 New Cross Rd., London, S.E.14. Chemists, druggists, etc. £3,000. Dirs.: A. L. Gardiner and P. J. Nottage.

Robert Howden (Wholesale) 12.12.58. 11 Fenchurch St., London, E.C.3. Chemists. £200. Dirs.: C. F. Castle and A. E. Marsden.

H. Tilsley Ltd. 15.12.58. 486 Fulwood Rd., Sheffield 10. Chemists and druggists. £3,000. Dirs.: E. and Mrs. F. E. Fell and H. Tilsley.

Vanity Fayre (Holborn) Ltd. 15.12.58. 14 Sicilian Ave., London, W.C.1. Dirs. in medicines, chemicals and toilet requisites. £1,000. Dirs.: Beatrice A. Bacon and C. T. Weiss.

Albemarle Chemists Ltd. £1,000. Sub.: L. P. Ney, Adelphi Terrace House, 3 Robert St., London, W.C.2.

J. H. P. Greenwood (Chemists) Ltd. 8.1.59. 23 Barrett Road, Birkdale, Southport. £100. Dirs.: J. H. P. and Gwendoline A. Greenwood.

Chemical Research (Horticulture) Ltd. 8.1.59. 5 Gt. Winchester Street, London, E.C.2. £100. Dirs.: Eileen Pearce-Smith and E. S. Smith.

R. V. May (Chemists) Ltd. 9.1.59. 24 College Street, Burnham on Sea. £500. : R. V. and Mrs. M. E. May.

E.E.C.I. Ltd. 28.1.59. 18 Fleet Street, London, E.C.4. Mnfrs. of and dlrs. in chemicals, gases, etc. £100. Dir.: Dorothy M. Evans.

Crownfields Drug Co. Ltd. 28.1.59. £100. Dirs.: Mrs. I. M. McIlroy, 271 London Road, Romford, and Mrs. N. Butterworth.

North London Pharmaceutical and Trading Co. Ltd. 30.1.59. 42 Kilburn High Road, London, N.W.6. £100. Dir.: W. T. Watson.

K. J. Leather (Chemist) Ltd. 30.1.59.
13 Long Street, Middleton, Manchester. To enter into an agreement with K. J. Leather for the acqn. of the bus. cd. on by him at Manchester; to cy. on the bus. of chemists, etc. \$1,000. Dirs.: K. J. and Joan T. Leather.

Lee-Smith Chemicals Ltd. 3.5.59. Rosslyn Avenue, East Barnet, Herts. £100. Dirs.: J. Gordon, Mrs. D. E. Beggs, and Mrs. F. Milborne.

J. M. Lichtig (Chemists) Ltd. 30.12.58. 62 Stamford Hill, London, N.16. To take over bus. of chemist cd. on at Stamford Hill, N.16, by J. M. Lichtig, etc. £100. Dirs: J. M. and Mrs. F. Lightig.

Evansky Products Ltd. 31.12.58. 146 Bishopsgate, London, E.C.2. Mnfrs. of and dirs. in cosmetic, toilette and beauty preparations, etc. £100. Dirs: A. and R. Evansky.

W. R. Moore Ltd. 19.3.59. Loddon Close, Twyford, Berks. To enter into an agmt. with Muriel F. L. Moore; to carry on bus. of pharmaceutical and general chemists, etc. £100. Dirs.: to be appointed.

Bryland Chemists Ltd. 19.3.59. Gt. Portland St., London, W.1. £100. Dirs.: A. and M. Landy.

Western Aromatic Laboratories Ltd. 11.3.59. £2,000. Dirs.: D. and J. Lemon, Eastern Mansions, 24 South Rd., Weston-super-Mare.

Crendon Pharmacy Ltd. 11 rendon St., High Wycombe. 11.3.59. Crendon St., High Wycombe. £5,000. Dirs.: M. E. and M. A. Seymour. Koltown Ltd. 12.3.59. Chemists. £100. Dirs.: to be appointed. Sub.: T. A.

Herbert, 156 Strand, London, W.C.2.

Weed-Master Ltd. 13.3.59. of and dlrs. in insecticides, fungicides, weed killers, etc. £100. Dirs.: to be appointed. Sub.: W. D. Crane, 14-16 Fletcher Gate, Nottingham.

Ballard and Co. (Chemists) Ltd. 13.3.59. Market Place, Faringdon, Berks. £5,000. Dirs.: B. W. and E. M. Cook.

Harold Pocock (Chemists) Ltd. 16.3.59. 65 Cannon St., London, E.C.4. To take over bus. cd. on at 87 Holyhead Rd., Handsworth, Birmingham, by the late Harold E. Pocock, etc. £200. Dirs.: S. V. and B. I. Brown, B. J. Holland, L. K. Downing and J. C. Duncan.

Abbotsbury Laboratories Ltd. 16.3.59. 16 Shenley Rd., Boreham Wood, Herts. Analytical and consulting chemists, etc. Dirs.: A. J. P. and J. Martin.

Old Coulsdon Chemists Ltd. 17.3.59.

217 Coulsdon Rd., Old Coulsdon, Surrey. £2,000. Dirs.: J. C. and W. B. Mottram. Christopher White and Jones Ltd. 25.2.59. 40 New Bedford Rd., Luton, Beds. Chemists. £3,000. Dirs.: C. A. White and M. Jones.

Queensbury Chemists Ltd. 26.2.59. 57 Berkeley Rd., London, N.W.9. £1,000. Dir.: J. Badyan.

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Livingstone (Chemists) Ltd. 32-33 Commercial St., Leeds 1. £1,000. Dirs.: not named.

Calt Ltd. 23.3.59. 18 Essex St., London, W.C.2. Chemists and druggists. £100. Dirs.: not named.

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AMBAROBINS.—777,934. A. H.

ADRENOXYL.—B778,815; BECAN-TYL.—778,816. Société des Laboratoires Labaz.

TRACTINE.—778,967. Bayer Products

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LALON.—784,029. Lalon Ltd.

PLANISAN.—784,570. May and Baker Ltd.

IMBRETIL.—784,929. The Wellcome Foundation Ltd.

INDALITON.—785,389. J. R. Geigy S.A.

### NEW PATENTS

### COMPLETE SPECIFICATIONS ACCEPTED

#### **Pharmaceuticals**

Derivatives of the lysergic acid series and process for their manufacture. Sandoz Ltd. 811,964.

Process for the preparation of therapeutic magnesium aluminate hydrate. Byk-Gulden Lomberg Chemische Fabrik G.m.b.H. 810,816.

Derivatives of pyrimidine and the preparation thereof. Wellcome Foundation Ltd. (Burroughs Wellcome and Co.). 812.366.

Sulphonamides. Smith and Nephew Ltd. 811,522.

Hydrazides and thiosemicarbazones. Farmaceutici Italia S.A. 811,780.
Process for the production of cycloaliphatic 1.2-diamines and the resulting products. Geigy A.G. 810,844.

Diquaternary ammonium compounds and the preparation thereof. Wellcome Foundation Ltd. 811,643.

Chlorobenzimidazolone compounds. Parke, Davis and Co. 811,692.

Heterocyclic hydrazides. Farmaceutici Italia S.A. 811,816.

Process for the production of heterocyclic carboxylic acids of the pyrazolidine series as well as the esters and salts thereof. Geigy A.G. 811,700.

Geigy A.G. 811,700.

Acid derived from an alkaloid named deserpidine, its esters and salts thereof, and process for their manufacture. Ciba Ltd. 809,913.

#### Dyestuffs

Manufacture of compounds of the anthraquinone series. Ciba Ltd. 809,222. Copper-containing polyazo dyestuffs. Sandoz, Ltd. 809, 279.

Polyazo dyestuffs. Farbenfabriken Bayer A. G. 808,798.

Metallisable monoazo dyestuffs and their use. Geigy A. G. 808,919.

Tertiary amine oxides and their use in dyestuff preparations. *Imperial Chemical Industries Ltd.* 811,046.

Cyanine dyestuffs. Gevaert Photo-Producten N.V. 811,156. Disazo dyestuffs derived from diphenyl and their copper complex compounds. Farbenfabriken Bayer A.G. 812,102.

Metal complexes of dyestuffs of the benzene - monoaze - hydroxynaphthazole series, and their use. Geigy A.G. 810,745. Manufacture of a finely dispersed vat

Manufacture of a finely dispersed val dyestuff of the pyrenequinone series. Farbweke Hoechst Aktiengesellschaft Vorm. Meister, Lucius, and Bruning. 810,980. Water-insoluble azophthalocyanine dye-

Water-insoluble azophthalocyanine dyestuffs and process for their production. Farbenfabriken Bayer A.G. 811,222.

Metal complexes of monoazo dyestuffs. Farbenfabriken Bayer A.G. 813,186.

Process for producing water-insoluble azo-dyestuffs on the fibre. Farbærke Hoechst A.G. Vorm. Meister, Lucius, and Bruning. 813,986.

Monoazo dyes. Interchemical Corp. 813,906

#### **Antibiotics**

Penicillin salt and therapeutic preparations containing same. C. Pfizer and Co. Inc. 813,879.

Therapeutic compositions containing calcium acid novobiocin. Upjohn Co. 813,893.

Stable aqueous preparations of calcium amphomycin. Bristol Laboratories Inc. 813.896.

#### Herbicides, fungicides, etc.

Composition for the control of fungus organisms. American Cyanamid Co. 812,492.

Herbicidal or plant growth regulant compositions. Fisons Pest Control Ltd. 813,931.

Soil fumigant compositions. Dow Chemical Co. 812,677.

#### Steroids

Synthesis of steroids. Olin Mathieson Chemical Corp. 812,810.

Steroid compounds. Merck and Co. Inc. 814,069; 814,070.

Halogen-testanes and process for their manufacture. Ciba Ltd. 812,690. Steroid compounds and the preparation

thereof. Pfizer and Co. Inc. 814,000. Steroid compounds and method for the transformation of epoxy-ketones by the action of fermenting yeast. Farmaceutici

Italia S.A. 813,317.

17 - aromatic sulphonate - 16 - keto steroids. Lasdon Foundation Inc. 813,334.

17-aromatic sulphonate - 16 - hydroxy

steroids. Lasdon Foundation Inc. 813,335. Keto-steroids. Lasdon Foundation Inc. 813,333.

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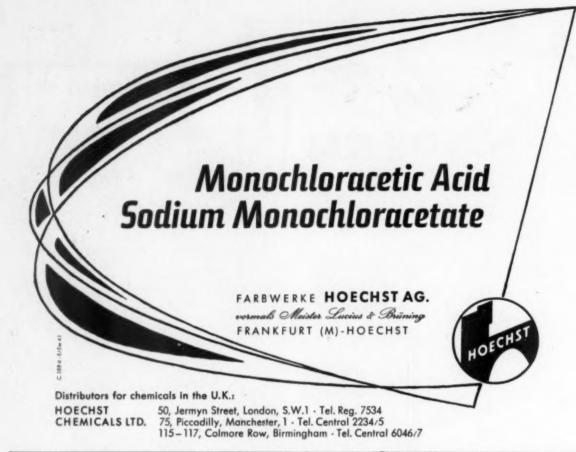
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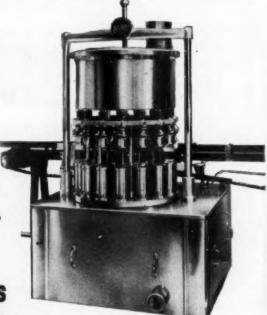
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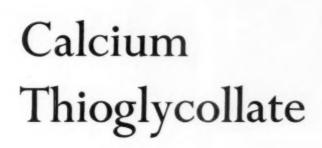
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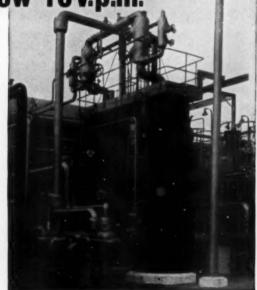
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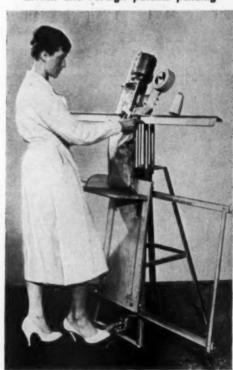
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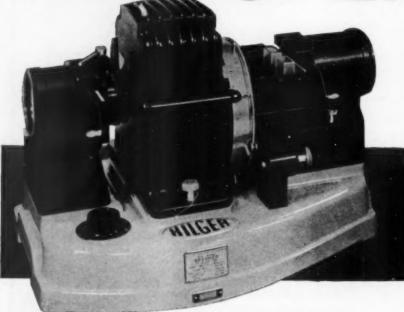
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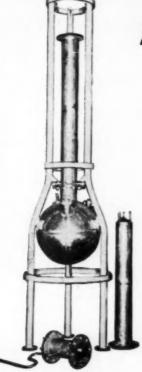
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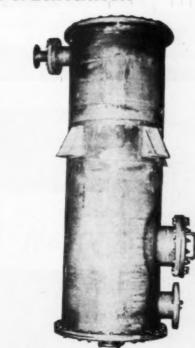


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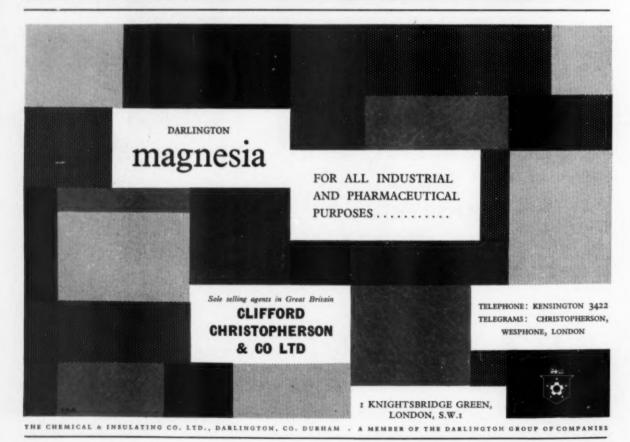
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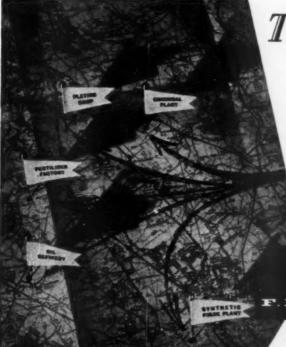
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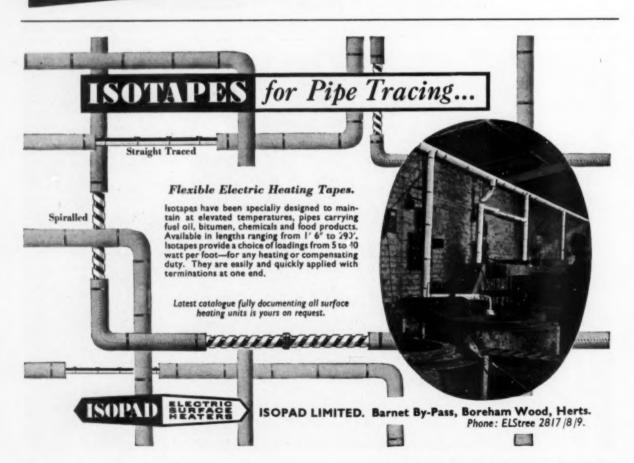
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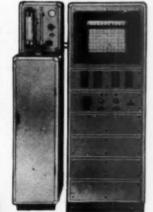
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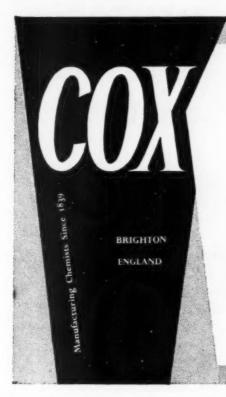
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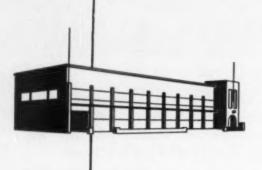


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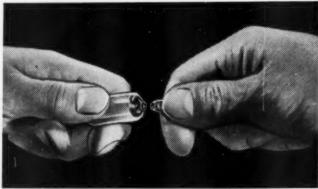
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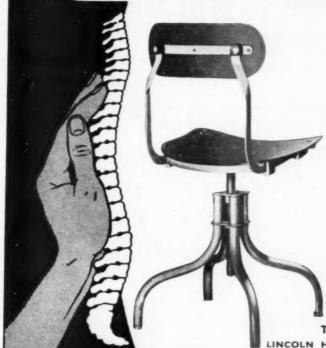
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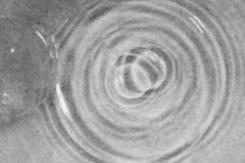
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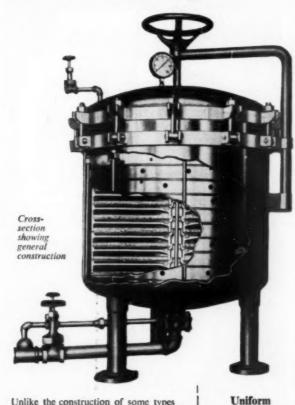
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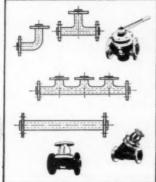
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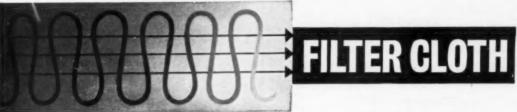
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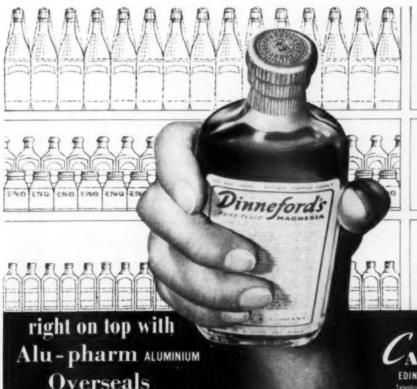
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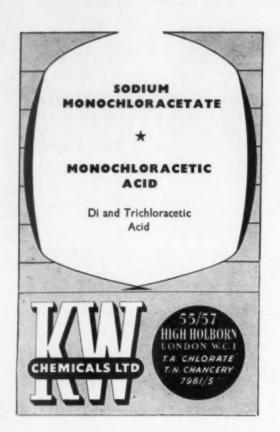


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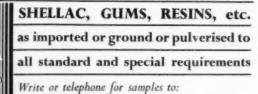
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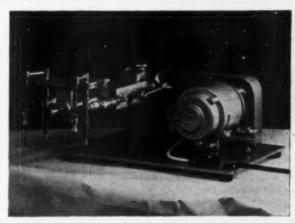
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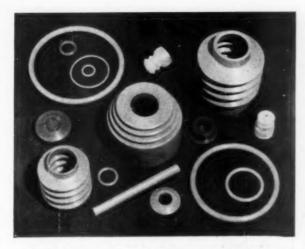


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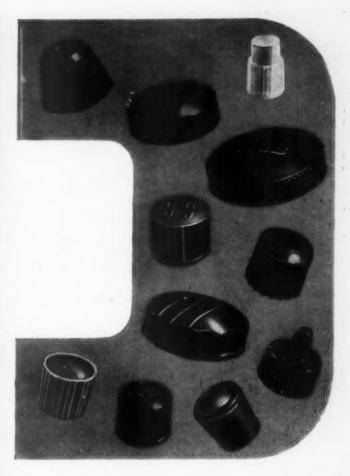
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